



## Official Notice.

### PATENTS, DESIGNS, AND TRADE MARKS (TEMPORARY) RULES, AUG. 21, 1914.

Until further notice, the following procedure will be adopted, under Rules 2 and 3 of the above Rules, in dealing with all work in connection with Patents, Designs, and Trade Marks (see this J., 1914, 813):—

1. During the continuance of the war no patent will be sealed and no registration of a trade mark or design will be granted to subjects of any State at war with His Majesty (hereinafter called "such subjects").

The term "such subjects," except for the purposes of paragraph 4 hereof, will be taken to include (a) a firm which by reason of its constitution may be considered as managed or controlled by such subjects or the business whereof is wholly or mainly carried on on behalf of such subjects; (b) a company which has received its constitution in an enemy's State; (c) a company registered in His Majesty's Dominions, the business whereof managed or controlled by such subjects, or is carried on wholly or mainly on behalf of such subjects.

2. As regards applications for patents, designs, and trade marks no distinction will in the first place be drawn between those made by such subjects and those made by other persons. All proceedings hereunder will be carried on as usual down to and including acceptance; but in the case of applications by such subjects all proceedings are suspended subsequent to acceptance (except such proceedings and matters as are mentioned in section 9 of the Patents and Designs Act, 1907) and will be suspended until otherwise directed.

The suspension of proceedings upon applications by such subjects will only be recalled or discharged upon proper terms including, if thought fit, the release of the applicant of the privileges and rights which he would otherwise have under Section 10 of the Patents and Designs Act, 1907.

3. Applicants who fail to conform to the provisions of the Patents and Designs Act, 1907, the Trade Marks Act, 1905, and the Rules made thereunder, will run the risk of losing their rights unless they are able to bring themselves under the provisions of Rule 3 of the above Rules. Applications under Rule 3 (a) should be made and will be considered at such time as the applicant, patentee, or proprietor of a design or trade mark as the case may be, is in a position to do the said act or file the said document as aforesaid. Applications under Rule 3 (b) should be made before the date for the doing of any such act.

4. As regards oppositions to the grant of patents and the registration of trade marks, arising after the commencement of the war, (a) oppositions by such subjects in cases where the grant or registration opposed is one to a British citizen or alien friend will not be entertained; (b) in the case where the grant or registration opposed is a grant or registration to any such subject the notice of opposition will be accepted, but all further proceedings will be suspended until the end of the war.

5. As regards inventions communicated by such subjects as aforesaid in respect of which patents are applied for by persons in this country, these will be treated in the same manner as if made directly by the communicator.

## Canadian Section.

Meeting held at Toronto, on Thursday, April 29th, 1915.

PROF. J. W. BAIN IN THE CHAIR.

### MANUFACTURE OF NITRITE OF SODA.

BY JAMES TURNER.

Of the various methods of converting sodium nitrate into nitrite, practical experience has shown that reduction by means of metallic lead is the method best adapted for working on a large scale. The reason is that both lead and saltpetre melt considerably below the temperature required for the reduction, and therefore the two substances can be brought into very intimate contact.

The transference of oxygen from the nitrate to the lead is slow and gradual, and, by proper regulation of the process, the amount of heat developed by the reduction is insufficient to destroy the nitrate completely; this may occur in the reduction with charcoal or calcium or barium sulphide.

Even in the lead process a percentage of nitrate is always converted into caustic soda, but in this case the by-products can be regenerated without difficulty. The lead oxide formed in the process can be converted easily and cheaply into lead.

The nitrite liquor formed in the lead process is so pure that the preparation of a salt containing a high percentage of nitrite presents no difficulties, while in all the other methods the liquors contain a considerable amount of impurity, making it difficult to prepare a pure salt.

The lead used must be a very pure soft lead, containing not more than about 0.2% of other metals. The lead used for sulphuric acid chambers is quite suitable. Less pure lead induces a violent reaction with consequent loss of yield, and produces too stiff a melt.

Commercial sodium nitrate is generally used, and should not contain more than 1% of sodium chloride, otherwise the melt is liable to become stiff. Recrystallised nitrate will give a better yield, but on account of the expense in recrystallising, it is preferable to use the commercial salt.

The reaction is carried out in a shallow cast-iron pan, which has been cast from the best pig iron procurable. Since the bottoms of the pans are heated to a dark red heat, and exposed to rapid changes of temperature, pans which are badly moulded, or cast from inferior iron, will soon crack or produce crevices, wherein the melt will lodge, and cause it to get overheated. The flue should be so arranged that the fire gases pass evenly around the pan.

The melt is mixed by agitators which extend across the pan, the teeth being so constructed that they clean or stir all the particles both from the sides and bottom of the pan. By continuous stirring, the lead is so divided that it offers a large surface to the nitrate. Good and uninterrupted stirring is therefore necessary for the rapid carrying out of the process.

Stirring is also essential because, at a temperature only a little above that at which the reaction between the lead and the nitrate takes place, lead

will act energetically on the pan, and the over-heated lead may cause a violent decomposition.

Again, lead oxide is a bad conductor of heat, and therefore, if it were allowed to remain for any length of time on the hot sides of the pan, over-heating might easily take place. On this account, the inner surface of the pan must be smooth. The middle of the bottom of the pan is so arranged that no oxide can deposit there.

The action of lead on melted nitrate commences at about  $420^{\circ}\text{C}$ ., and it is best to introduce the lead at that temperature. Sodium nitrate melts at  $330^{\circ}\text{C}$ . and lead at  $335^{\circ}\text{C}$ . The temperature is taken by means of a pyrometer, but an experienced man can also tell by the colour of the oxide formed, as well as by the rapidity with which the lead disappears, if the temperature is right.

The charge usually used for the melt pans is 275 lb. of nitrate and 672 lb. of lead. 250 lb. of the nitrate is put into the pan and heated to  $420^{\circ}\text{C}$ ., and then 2 cwt. of lead is added in bars of 25 lb. each. When all the lead has disappeared (which can be ascertained by stopping the agitators and taking a sample out of the middle of the pan) a further 4 cwt. of lead is added, 25 lb. at a time, care being taken that all the lead has been oxidised before making a further addition. This will require about 8 hours in all. When the final charge of lead has been oxidised, a further half hour's heating is given and the melt is finished. The temperature must be kept at about  $420^{\circ}\text{C}$ . during the whole operation. The percentage of nitrite in the melt may be ascertained by titration with permanganate.

The 25 lb. of nitrate is held in reserve, to be used for cooling the pan should it get a little overheated.

The melt is now run into water in a long, semi-cylindrical cast-iron box provided with a strong agitator running through the centre. At one end is a series of taps, at different levels, through which the clear liquors are run. At the end of this box are three cast-iron receiving tanks: No. 1 tank receives the strong liquors from  $30^{\circ}\text{B}$ . downwards, No. 2 tank weak liquors,  $5^{\circ}$  to  $6^{\circ}\text{B}$ ., and No. 3 tank receives the lead oxide. The washer is provided with strong iron covers, having a manhole through which the melt is poured. The washer must contain sufficient water to produce a liquor of about  $30^{\circ}\text{B}$ .

The washer is allowed to run for half an hour, and the contents are then allowed to settle. The clear liquor is run off into No. 1 tank, the washer is again filled with water and allowed to run for another half hour, and the liquor run into No. 1 tank, and this is repeated until all the nitrite is dissolved. All liquors down to about  $5^{\circ}\text{B}$ . go into No. 1 tank.

The weaker liquors are run into No. 2 tank, until the melt is free from nitrite, then the agitators are set in motion, water is allowed to run in continuously, the outlet from the washer is opened, and the water is run into No. 3 tank; this washing is continued until all the lead oxide is washed out of the melt. The residue in the washer consists chiefly of metallic lead and higher oxides of lead, and is run into a receiver beneath the washer.

The liquors from No. 1 tank are treated with a little nitric acid to neutralise any caustic soda which has been formed during the melt, and are then blown into the evaporators. The liquor in No. 2 tank, which contains very little nitrite, is used in place of water for washing the next melts.

The evaporators are made of wrought iron and are provided with steam coils. The evaporation is continued until the liquor reaches  $44^{\circ}\text{B}$ ., when it is allowed to settle, and then run into crystallising tanks and allowed to crystallise for several days according to the conditions of the weather.

The best crystallising tanks are made of cast iron, about 12 ft. long, 6 ft. wide, 2 ft. deep.

When crystallisation is finished, the liquors are run off and the crystals are taken from the sides and the bottom and piled up in the centre of the tank to drain; they are then centrifuged and dried in a stove. The product contains about 96 to 98% of sodium nitrite. The mother liquor from No. 1 crystals is evaporated to about  $48^{\circ}\text{B}$ . and the liquor crystallised, yielding nitrite of  $85\%$  to  $90\%$  purity.

The liquors from No. 2 tank are evaporated to about  $50^{\circ}\text{B}$ . to produce a  $70\%$  nitrite. The liquors from No. 3 tank are evaporated to dryness and the residue, which contains about  $50\%$  nitrate, is used again in the melt pan.

The mass of lead and higher oxides which is removed from the washers is melted and the lead is run into moulds, holding about 25 lb. each, and is used over again.

The lead oxide is removed from No. 3 box in the form of paste, and is dried slowly on a kiln, ground, and dressed; it is put on the market as litharge.

By careful regulation of the above process,  $96\%$  of the theoretical yield of nitrite may be obtained.

A process was patented in 1896 (Eng. Pat. 1743) by Dr. Paul and Messrs. Read Holliday and Sons, Ltd., for the reduction of sodium nitrate by means of caustic soda and sulphur. This process has not proved to be so cheap as the lead process, on account of the difficulty in separating the sodium sulphate from the nitrite, and the maximum yield is only  $80\%$  of theory.

In this process, 2200 lb. of sodium nitrate and 380 lb. of caustic soda are melted by open fire in a large cast-iron pan fitted with mechanical stirres; the flue is constructed to allow even distribution of heat around the pan. When the whole is melted, the agitators are started, the temperature is raised to  $400^{\circ}$  to  $420^{\circ}\text{C}$ ., and 120 lb. of sulphur is added very slowly in small quantities. A further 60 lb. of melted caustic soda is then added, and another 20 lb. of sulphur, and these additions are repeated until a total of 896 lb. has been added, including the 380 lb. of caustic which was put in originally and 300 lb. of sulphur. This process usually occupies about 8 hours.

If the sulphur is added too rapidly, serious explosions may occur; even with the most careful working it is impossible to avoid some explosions, due, no doubt, to organic matter coming in contact with the sulphur and nitrate. If the temperature of the melt is allowed to rise too much, the sulphur will be oxidised.

The sulphur is added to the melt by means of a very long iron rod, with flat end, which is manipulated from behind an iron partition.

When the melt is finished, as shown by testing it with permanganate, it is allowed to run for a further hour or so, and is then transferred to a wash box of similar construction to the melt pan, but having a wrought-iron cover. The wash box must contain sufficient water to produce a clear liquor of about  $50^{\circ}\text{B}$ .

As sodium nitrite is more soluble than the sulphate, a large percentage of the sulphate crystallises out, and is separated by means of a vacuum filter covered with burlap, the clear liquor being run into a boiler. The sulphate, which contains a certain percentage of nitrite, is washed once or twice with water, and the wash-water is used in place of water for the next melt. The liquor in the boiler is evaporated to about  $44^{\circ}\text{B}$ .; during this process a large amount of sodium sulphate separates and is removed at intervals by means of long shovels, and put into perforated wooden boxes placed over the evaporators, so that the liquor can drain back. The concentrated liquor is allowed to settle and run off into the crystallisers, and the mother liquors are worked up as in the lead process.

The reaction involved in this process is:—  
 $3\text{NaNO}_3 + 2\text{NaOH} + \text{S} = \text{Na}_2\text{SO}_4 + 3\text{NaNO}_2 + \text{H}_2\text{O}$ .

## Glasgow Section.

Meeting held at Glasgow on Tuesday, 20th April, 1915.

MR. ROBERT HAMILTON IN THE CHAIR.

## A NEW MACHINE FOR THE PREPARATION OF VULCANISED RUBBER FOR ANALYSIS.

BY R. WHEATLEY, B.SC. (LEEDS), A.I.C., AND B. D. PORRITT, B.SC. (LOND.), F.I.C.

When it is required to examine chemically many samples containing rubber as a constituent, the preparation of the material for analysis necessitates special consideration and occupies a considerable amount of time.

In the preparation of any material for analysis the sample must comply, so far as is practicable, with the following essentials:—

- (1) It should represent accurately the bulk.
- (2) It should be so prepared that it is thoroughly and uniformly acted on by the reagents subsequently employed.
- (3) Its preparation should be as rapid and simple as possible, necessitating the minimum amount of skilled supervision and time.

The methods at present available for the preparation of vulcanised rubber cannot be considered as generally applicable or entirely satisfactory when examined from the above standpoints. The explanation for this must be sought in the wide variation in physical properties of the different types of manufactured goods in which rubber forms a constituent, and the differing processes of manufacture and vulcanisation. The material may vary enormously in character from elastic thread to vulcanite; it may be tough, as in the case of band tyres, or crumbly, like an eraser, and may be bulky or flimsy, as exemplified by railway buffers and cut sheet.

A further complication is introduced by the fact that rubber frequently occurs in combination with textile fabrics, from which its separation is often exceedingly difficult and can at the best be effected only imperfectly.

The general analytical processes employed for the chemical evaluation of rubber goods are designed to ascertain the various additions made to the rubber, whether mineral or organic matter, and generally comprise ash determination by gentle incineration, Soxhlet extraction with acetone, and treatment with alcoholic potash to remove saponifiable organic matter, together with the total sulphur determination.

In the preliminary selection of the sample the following points should be kept in view:

- (1) Rubber may retain a considerable amount of moisture, necessitating preliminary drying, which must be carried out so as to preclude the possibility of the formation of resins by oxidation or the loss of sulphur by heating.
- (2) On account of the adhesive character of the vulcanised material, it is customary to dust the surface with French chalk during the manufacturing processes. An undue proportion of surface material will therefore probably result in an excessive result for mineral matter.
- (3) In steam- and press-cured articles, sufficient free sulphur is usually present to migrate and "bloom" on the surface. Neglect of this fact may lead to a considerable error in the determination of sulphur.
- (4) A varnish or surfacing is employed in certain instances, for example, goloshes and surgical goods, and should be removed before the sample is prepared for analysis.

(5) More than one quality of rubber compound may be employed in making up an article, for instance, the sole, heel and upper of a shoe, and the tube and cover of a hose.

(6) The raw material may not be uniformly mixed and the vulcanisation in thick articles may not be uniform.

Careful selection and preparation of the sample, fine division, and thorough mixing are therefore necessary to ensure reliable analytical results. The great variation in physical properties, shape, and character of rubber goods, however, renders it unlikely that any one device for the reduction of samples to a fine state of division will be uniformly satisfactory. Up to the present the problem has, therefore, received a considerable amount of attention.

Henriques, in his pioneer work on rubber analysis, alludes to the difficulties involved in the preparation of an average sample, and recommends taking numerous sample strips and cutting these into small pieces.

Weber\* recommends the employment of a small laboratory mixing mill with uneven speed rolls to grind the material to a flaky condition. He states, however,† "that if the sample consist chiefly of rubber with but little foreign admixture, it cannot be 'crumbed' by filing, and even on the mixing roller may occasionally refuse to break up, forming sheets instead."

Later writers‡ recommend the use of uneven speed mixing rollers followed by grinding in a mortar or coffee mill when dealing with well-cured samples containing about 50% of mineral matter. Ebonite and soft mixings, which they note as failing to respond to this treatment, must be rasped with a coarse file in the one case and in the other finely chopped with scissors.

Schidrowitz draws attention to the fact that oxidation may take place, with the production of resinous matter, if the sample is subjected to long continued mechanical abrasion at high temperature.

A convenient type of laboratory mixing roll of this kind for the preparation of rubber samples is fully described by Archbutt in a recent publication.§

It has been proposed to take advantage of the fact that rubber becomes brittle at low temperatures, by freezing the samples in liquid air and then powdering in a mortar.¶ Owing to the rapidity with which the frozen rubber warms up, passing from a brittle to an exceedingly tough condition, and the fact that liquid air is not available to the average analyst, this suggestion cannot be considered generally practicable.

As a result of experience in dealing with a large variety of samples of different qualities, it was decided that rasping was in general the most satisfactory method for the preparation of material for analysis. This is, however, a very tedious process, and it seemed that some mechanical method of abrasion might be adopted in order to simplify the operation. The methods employed in the factory to "buff" rubber goods were considered, and of these a high speed carborundum wheel and a high speed circular steel toothed cutter seemed to give the most promising results.

The employment of carborundum as an abrasive for this purpose was abandoned, owing to the danger of its loss and the consequent contamination of the sample when dealing with vulcanite and heavily compounded qualities, and we endeavoured to modify the arrangement by substituting a circular flat file mounted on the

\* Chemistry of Rubber, p. 237.

† Chemistry of Rubber, p. 227.

‡ Schidrowitz' Rubber, p. 255, and Potts' Chemistry of Rubber Industry, p. 121.

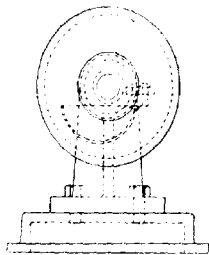
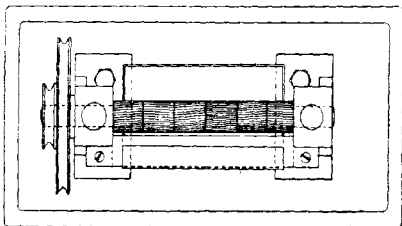
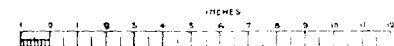
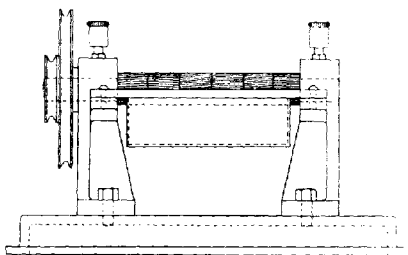
§ Analyst, 1913, 38, 550 (see this J., 1914, 34).

¶ Potts' Chemistry of Rubber Industry, p. 53.

rim of a wheel. File-makers condemned the design as impracticable, and on reconsideration the present apparatus was constructed.

The abrasive surface consists of a circular file,\* made from a 1-inch diameter rod of file steel, the cutting edges being parallel to the axis. This is mounted in bearings set on a bed plate, and has a double pulley keyed to one end. A small electric shunt-wound motor, of one-eighth H.P., furnishes the necessary motive power to drive the file at 1200–2400 revolutions per min., according to the pulley wheel used. These rotational speeds correspond to peripheral speeds of 314 and 628 feet per minute, respectively.

The rasped rubber falls into a detachable tray placed below the file and supported by a bar running between the brackets.



With this apparatus small vulcanised rubber samples, varying from hard vulcanite to soft pure qualities, may be rapidly and effectively reduced to a fine state of division. For the harder qualities the low speed gives the more satisfactory results, while when dealing with soft material a better result is obtained by increasing the rate of revolution.

As a rise in temperature takes place during the operation and the resultant material is in a finely-

divided condition, it was thought desirable to ascertain whether any alteration took place in the acetone extract, due to oxidation of the rubber or volatilisation of sulphur. Experiments have been made to test this point, and the composition of the rubber was found to be unaltered by the treatment.

With the material in a finely-divided condition there is no difficulty in obtaining a representative sample for analysis.

The acetone extraction is rendered more complete, and the alcoholic potash extraction is made more reliable owing to the state of division being uniform and permitting more thorough penetration by the reagent. This, however, results in certain cases in an increased loss, owing to solution of mineral matter.

From the results of our experiments we draw the following conclusions:—

(1) Small samples of vulcanised rubber may be reduced to a finely-divided condition without alteration in composition.

(2) Care must be taken to protect the finely-divided material from oxidation if not immediately used for analysis, or if preliminary drying is required.

(3) Care must be taken to prevent oxidation during the course of analysis.

It would appear therefore that the reduction of the material to a fine state of division, with the resultant advantages of uniformity of composition and efficiency of extraction, is accompanied by the risk of oxidation.

The present apparatus is ineffective in the case of raw rubber and pure reclaimed rubber; possibly a considerable increase in the speed of revolution might admit of its employment in this field, but, on account of the adhesive nature of the material, the particles would tend to coalesce during subsequent analytical operations and render the preparatory treatment valueless.

## Manchester Section.

Meeting held at the Grand Hotel, on Friday, March 5th, 1915.

MR. JULIUS HÜBNER IN THE CHAIR

## THE CONCENTRATION OF SEWAGE SLUDGE.

BY J. GROSSMANN, M.A., PH.D., F.R.S.

The disposal of sewage sludge has for a considerable time engaged the attention of those who are interested in the purification of sewage. There is now no difficulty in obtaining a satisfactory effluent, but whatever method of dealing with sewage may be used, the problem of disposing of the sludge has to be taken into consideration, and in the solution of that problem the concentration of the sludge plays an important part.

In general, there should be no difficulty in obtaining a sludge from the settling tanks which contains on an average about 10% of solid and 90% of liquid matter. There are isolated cases of troublesome sludge, owing to local trades, which will not settle to a consistency of more than 5% solids; but in most cases where such abnormally weak sludge is obtained, it is due either to a faulty construction of the tanks, or to faulty work in connection with the emptying of the tanks and the removal of the sludge.

\* Makers: Messrs. Alex. Mathieson and Sons, Ltd., Salford Tool Works, Glasgow.

It is evident that even in the simplest modes of disposing of the sludge, which consist in dumping it either on land or into the sea, the concentration of the sludge will affect the cost of disposal very materially.

It is specially important to obtain sludge in the most concentrated form from the settling tanks in those cases where the sludge is carried in boats to the sea, as will be shown further on.

Even where the sludge is submitted to filter-pressing, its original concentration is of some influence on the length of the operation, and therefore to some extent on the cost of the process. I have stated in a previous paper that where lime is used, the most careful experiments show that, taking everything into consideration, a cake containing about 30% of actual dry sludge will cost about 2s. 4d. per ton to produce; calculated on the ton of actual dry sludge, that would come to about 7s. 9d. per ton. If no lime were to be added to the sludge, the cost of filter-pressing would not only be greatly increased, but it would be extremely difficult by that means to get uniform sludge containing 30% of actual dry matter. In many processes dealing with the utilisation of sludge the presence of lime is objectionable and it, therefore, in order to prepare a concentrated sludge for these processes it were necessary to filter-press without lime, considerable additional expense would be incurred.

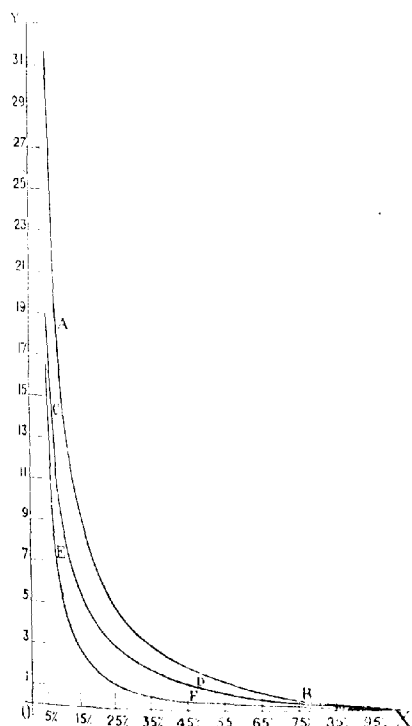


FIG. 1.

Wherever it is desirable to reduce sewage sludge to a state of dryness by means of heat, the amount of water contained in it will determine directly the amount of fuel required for drying, but there is a limit beyond which it may not be worth while to abstract water by purely mechanical means.

In general, if  $x$  is the percentage of solid matter and  $y$  the amount of water in tons to be evaporated to yield one ton of dry sludge,  $y = \frac{100}{x} - 1$ . This

represents the curve, CD, which is an equilateral hyperbola, with OY as one asymptote and the other parallel to OX but raised up 1 from it, and in which the percentages of solid matter are plotted on the abscissa and the amounts of water to be evaporated on the ordinate. This curve shows that down to a certain point the differences are enormous, and beyond that point they are almost proportionate to the percentage. Suppose that in a properly constructed drying machine one pound of coal will evaporate 6 lb. of water from sludge, and suppose the cost of coal is 10s. per ton, we can plot a curve which shows at once the cost of drying sludge of different percentages. The general formula to this would be as follows:—

If  $m$  = price of coal in shillings per ton;  $n$  = lb. of water evaporated per ton of coal, and  $z$  = cost of evaporating sludge containing  $x\%$  of solid matter to dryness,

then  $z = \frac{m}{n} \left( \frac{100}{x} - 1 \right)$ , and this is represented by the curve AB, in which  $m = 10$  and  $n = 6$ .

If  $x$  equals the solid matter in sludge of a certain concentration,  $x'$  the percentage of solid matter in another sludge of different concentration, and  $d$  the extra cost in evaporating the weaker sludge as compared with the more concentrated sludge,

then  $d = \frac{100}{x} \left( \frac{x - x'}{x'} \right)^m$ ; this is represented by the curve EF, in which  $m = 10$  and  $n = 6$ .

Fig. 1 shows the curves AB, CD, and EF, and the numerical data belonging to them are given in the table.

$x$ Percentage of solid matter.	$y$ Tons of water to be evaporated to produce 1 ton dry.	$z$ when $m=10$ and $n=6$ Cost of fuel in shillings at 10s. per ton of coal, 6 lb. water per lb. of coal.	$d$ Differences in shillings in cost.
5	19	31.667	
10	9	15.000	16.667
15	5.667	9.444	5.555
20	4	6.667	2.777
25	3	5.000	1.667
30	2.333	3.889	1.111
35	1.857	3.095	0.794
40	1.50	2.500	0.585
45	1.22	2.037	0.463
50	1.00	1.667	0.370
55	0.82	1.364	0.303
60	0.67	1.111	0.253
65	0.54	0.897	0.214
70	0.43	0.714	0.183
75	0.333	0.556	0.158
80	0.25	0.417	0.139
85	0.176	0.294	0.123
90	0.111	0.185	0.109
95	0.053	0.088	0.097
100	0	0	0.088

It will be seen that whereas the difference in the cost of drying a sludge of 5% and 10% solid matter amounts to 16s. 8d. per ton, the difference in drying sludge of 30% solid matter and 35% solid matter only amounts to about 7d. per ton. If, therefore, it would require special apparatus to reduce a 30% sludge to 35%, unless all the costs incidental to this would be less than 1s. per ton of dry matter (which means 2d. per ton of 30% sludge) it would not be worth while to have recourse to that extra operation. The general conclusion to be drawn from these considerations is that on the whole it will not pay to reduce the sludge by purely mechanical means to more than 30% if it has to be subsequently dried by heat. A sludge of 20% even would be economical to dry, and anything

between 20% and 30% would give highly satisfactory results.

These considerations led me to endeavour to find some means by which I could obtain a sludge of 20% or more solid matter by ordinary mechanical means. It is well known that sludge of that consistency can be obtained by draining on sand or cinder beds, but apart from the fact that the sludge obtained would vary considerably in concentration, it would, after draining, have to be removed by manual labour, which is an extremely costly operation, more costly even than that of filter-pressing. It became evident that whatever operation was designed would have to be one which could be carried on automatically by machine power. I constructed a tank on these lines which contained a filtering compartment through which the sludge, which settled in the main part, could drain its superfluous water. After as much of the water as could be drained off had been removed, the concentrated sludge was emptied out of the settling tank by means of a conveyor screw placed at the bottom of the tank and worked from an engine. This arrangement gave fairly satisfactory results, in so far as it was possible to concentrate sludge from 10 or 12% solid matter, or more, to about 17 to 20% of solid matter, but the time occupied was too long. In order to produce this effect the sludge had to settle for a week, and during that time was liable to become partially septicised; cavities would then form in different parts of the mass of sludge and as it is impossible to avoid this septicising of the sludge, which may occur not only in summer but at other times of the year, according to the state in which the sludge leaves the settling tanks, the results obtained in this manner were frequently disappointing. In working drying machines by a continuous and automatic process, which is the only way in which they can be worked where large quantities have to be dealt with, it is of the utmost importance to have material to deal with which is of uniform strength, and the variations produced by the septicising of the sludge were such as, at times, to cause considerable inconvenience to the working of the dryers.

The method which I have described is one in which the sludge settles to the bottom whilst the clear top liquor is automatically drained off through the filtering chamber, i.e., it is a method of settling downwards by gravitation. The question arose whether it would not be possible to find a practicable means of separating part of the water contained in the sludge in such a manner as to make the sludge, so to speak, settle upwards instead of settling downwards, and I found that this object could be attained by means of sulphuric acid. The first experiments were carried out in the laboratory, and I discovered that if sludge from the settling tanks was mixed in the proportion of 1000 parts of sludge to 3 parts of sulphuric acid which had been diluted with water to yield about a 10% solution, and the mixture left standing, a gradual separation took place. At first, after standing for one day, there was a slight amount of almost clear liquor at the bottom, and this separation went on gradually until (after three days) practically one-half of the mass at the bottom was clear liquor, whilst the other half at the top was sludge of nearly double the concentration of the original 10% sludge which had been used for the experiment. It was further found that the sludge thus obtained drained better than the original sludge, and work carried out in the tanks which I had originally designed for the downward settling process, showed that no septic action took place after the treatment with sulphuric acid.

This method for concentrating sludge has now been in use at the Oldham Corporation Sewage Works for more than twelve months, with invariably satisfactory results, and altogether about 20,000 tons of sludge of 10% solid matter has

been treated by it. The composition of the sludge entering the tanks varies from 10 to 13%, and the resulting product fluctuates between 19 and 21% of solid matter. The sludge from the sedimentation tanks is pumped into the patent settling tanks, and in the process of pumping a quantity of sulphuric acid of 148° Tw. (sp. gr. 1.74), previously diluted with about ten times its volume of water, is mixed with it, and the resulting mass left to stand for three days; after that time, the concentrated sludge has separated at the top and the clear water, which is below, is run off through a suitable arrangement of taps. The sludge is left for 24 hours in the tanks for draining, and is then removed by a conveyor screw placed at the bottom of each tank and taken straight from there by means of a bucket elevator into the hoppers of the drying machines, where it is reduced to dryness by means of direct heat. The total cost of the process, calculated per ton of sludge containing 10% dry substance, is as follows:—

Cost of sulphuric acid .....	0.064s.
power and labour .....	0.074s.
Sinking fund charges .....	0.050s.
	0.208s.

—2.496d. = 25d. per ton 10%.

Calculated on dry substance, it is, per ton, as follows:—

Cost of sulphuric acid .....	1.04s.
power and labour .....	0.74s.
Sinking fund charges .....	0.50s.
	2.28s.

At the beginning of this year I made further improvements in the construction of my tanks by which I obtain a sludge, on an average, of 26% solid matter. The cost of obtaining 26% sludge is 6½d. per ton.

By adopting a slightly different mode of working, I could obtain a sludge of even higher concentration, up to 30% solid matter, but for the purposes of subsequent drying, there would not be sufficient advantage from a commercial point of view in working for the obtaining of such a concentrated sludge. For the purpose of comparison, it may be stated that the cost of producing sludge of 30% solid matter would be somewhere about 7½d. per ton. As a sludge of this concentration would be equal to the average sludge obtained by filter-pressing without the use of lime, which would cost about 4s. per ton at the least, it is evident that by using my process and apparatus, a saving of over 3s. per ton could be effected, which, in a town of 100,000 inhabitants, would amount to £1,000 per annum.

Equally striking are the figures which we obtain on comparing the present cost of carrying sludge by steamer to the sea with that resulting from the adoption of my concentration process. The average concentration of sludge carried to the sea may be taken at 12½%; it is evident that by concentrating that sludge to 26%, its bulk would be reduced more than half, so that a steamer could take in one journey as much as it now takes in two. Apart from the saving in freight charges, there would be a further advantage in places like Manchester and Salford. In these places the steamers in use at present are worked to their fullest capacity and cannot cope with the quantity of sludge which has to be removed. The adoption of my method would do away with the necessity of buying additional steamers which, as they could only be partially employed for some considerable time, would add very greatly to the present cost of sludge removal. The cost of sludge removal in Manchester at present is 9½d. per ton of 12½%; by concentrating this sludge to 26%, the cost would be reduced to 7.68d. per ton. This on a yearly output of 200,000 tons would effect a saving of £1500 per annum.

The most important results of the concentration of sludge by the means which I have indicated will, however, be found in those processes of sludge disposal in which as an incidental part of the process sludge has to be reduced to dryness by heat. It may be said that the preliminary concentration by mechanical means is and will be the fundamental basis of all processes which aim at the production of a material from sludge which is to be used for fertilising purposes, and it is on this account that I have given such special attention to the subject.

I have shown above that where sludge has to be reduced to dryness, it makes little difference in the cost whether we start with a sludge containing 25% solid matter or 30%, but what is of the utmost importance is to ascertain what it costs to reduce the original sludge of 10 or 12½% to 25%. If we compare the different methods which might be used for reducing the sludge to that consistency, we find that in order to produce ultimately one ton of dry sludge it would cost about 12s. to 15s. at least to do this by means of filter-pressing, considerably over 20s. by spreading on filter-beds, and somewhat about 2s. by means of the sulphuric acid process. As the further cost for fuel of producing one ton of dry sludge would be about 5s., the cost of the two items in using my process would be about 7s., whilst the lowest cost in using any other process would be close on 20s. per ton.

It is satisfactory to note that more attention has lately been given to the great importance of utilising the manurial properties contained in sludge. Of course it is evident that as sludge is poor in phosphates and potash salts, it can never be used as a complete manure, but, on the other hand, if properly treated, and if free from grease (which is a highly objectionable constituent), it has been found in practical work to be an extremely valuable fertiliser base on which to build up so-called concentrated manures. Whatever process may be used for producing a fertiliser base from sewage sludge, it is certain that it will necessitate the reduction of the sludge to dryness. Even if there were no other reasons for drying the sludge, the fact that the cost of carriage plays an important part in the manure trade would necessitate it. To the best of my knowledge the method which I have designed for the preliminary elimination of water by mechanical means is more efficient and more economical than any other known method. Even where the sludge is not intended for manurial work but is dumped on the land, it will be found far cheaper to use the settling process which I have described and, if necessary, mix some cinders with the settled product to stiffen it before placing it on the land, than to use either filter-pressing or draining on cinder beds; but it is to be hoped that this enormous waste of material which should go back to the land, and which represents a value of at least £2,000,000 per annum in this country, will not continue indefinitely, and that it will be recognised that sewage sludge is a national asset which should be dealt with by the Government in the interests of agriculture, to which a cheap and efficient manure will be of incalculable benefit.

#### DISCUSSION.

Mr. F. R. O'SHAUGHNESSY considered that sludge treatment by means of sulphuric acid was an extremely ingenious and interesting process, though he felt doubt as to the results obtainable on a large scale. Some years ago he had conducted experiments with regard to the concentration of sludge by direct gravitation. A tower 20 feet in height and approximately 5 feet in diameter, with apertures at intervals of 2 feet, was used. Experiments were made both with crude sludge and septic sludge, and the results were broadly in agreement with those obtained by Dr. Grossmann at Oldham. With a sludge of

about 10% solid matter, a period of four or five days was required to arrive at the point of maximum concentration of the column of about 83 or 84% water, or about 16 or 17% of solid matter. Also it was found that with the septic sludge used, after the third or fourth day the effect of gravitation was reversed, owing to the development of gas, when the whole operation was ruined. In the end the process did not appear to be practical, though of scientific interest. One very important item, in connection with the treatment of sludge on a large scale by sulphuric acid, was the liquor which was drained off. Dr. Grossmann's diagram indicated that the experiment had worked excellently in the laboratory, but when dealing with tens of thousands of tons of highly acid liquid per annum, a very considerable problem remained to be solved. In concentrating sludge by mechanical means one had a very large quantity of liquor which contained an appreciable quantity of solid matter. Frequently it was convenient to pass the liquor again through the tank system. The whole question was one of economics when one was preparing solid matter for ultimate use in some other process. In a paper read at the International Congress of Applied Chemistry, London, in 1905, Dr. Grossmann referred to 50% concentrated cake when the sludge was pressed, and also to the point that the nitrogenous matter in the sludge was still in the unconverted state, being only very partially affected by the sulphuric acid. That, of course, was a most important matter. There was an enormous loss of nitrogenous matter when it was actually put on the land. He asked what were the actual results obtained in the field, in the case of large-scale experiments, with the products obtained by means of the process under discussion. He could not agree with Dr. Grossmann that the cost of drying sludge on the land would be about 20s. per ton. At the Birmingham sewage works the drying process was actually carried on on a large scale, dealing with hundreds of thousands of tons, for very much less than 20s. per ton. They did not, however, dig the sludge into the land.

Mr. DRUCKWORTH thought Dr. Grossmann was modest in allowing 6 lb. of water per lb. of coal. Personally, he was of opinion it would be quite possible to obtain 7 or 8 lb. by evaporation under pressure.

Mr. E. ARDERN agreed that the question of sludge concentration was entirely one of cost and that the comparative costs given for alternate methods of sludge concentration must be accepted with some reserve. In a recent publication by Kershaw, information collected by the Royal Commission on Sewage Disposal was given in tabular form, from which it would be seen that for a representative group of ten towns the average cost of pressing sludge (including cost of lime and capital charges on plant) was 6d. per ton of wet sludge dealt with. It could be assumed that with efficient pressing the moisture content of the sludge would be reduced from 90% to from 50 to 60%, which figure could be compared with the cost given by Dr. Grossmann for reducing the moisture content from 90% to 80% by acid treatment. It was also necessary to know whether the actual volume of sludge was reduced proportionately to the amount of water separated or removed by acid treatment. In his experience this was not the case, owing to the frothing action of the acid, and consequently the removal of say 50% by volume of water did not necessarily mean a reduction by one-half of the sludge to be dealt with. Where acidification was required in the subsequent treatment of the sludge, e.g., in the recovery of grease, etc., he could quite understand the advantage of the preliminary addition of a



small proportion of the total acid required for the purpose of concentration prior to drying or distillation.

Mr. P. GAUNT enquired as to the application of the process, where sludge was disposed of by ploughing into land. Evidently the acid exercised a certain sterilising effect, since septic action was inhibited. How would this affect the subsequent disintegration of the sludge upon the land? Usually a fairly high lime content was maintained in land used for this purpose. A sludge which had been acidified even to a slight degree might prove more resistant to the desired disintegration.

Dr. T. J. I. CRAIG asked Dr. Grossmann what he considered the best method of obtaining the sludge most suitable for the treatment dealt with in the paper. Another point was what became of the sulphuric acid? Was the liquor left acid or was it neutralised in any way by the sludge? Did the addition of acid to the sludge dissolve any valuable constituents such as phosphates?

Dr. BENTLEY inquired whether Dr. Grossmann had tried sodium bisulphate in his process. It was conceivable that a use might be found for this by-product by means of Dr. Grossmann's process.

Mr. S. EVANS asked how much clear liquor was decanted from the bottom of the tank. He suggested that the action might be similar to that which occurred in the separation of some minerals in ore-floatation processes.

Dr. GROSSMANN, in reply, said that the majority of the speakers had referred to the large quantities of acid used, but, as a matter of fact, the acid used specifically for the separation of the sludge was very small, and amounted only to 3 parts  $H_2SO_4$  per 1000 parts of sludge. The acid became practically neutralised, and the liquor which left the tanks was so slightly acid that it was difficult to ascertain its acidity. He wished particularly to emphasise the fact that the results given were not obtained from laboratory experiments, as some of the speakers assumed, but represented facts ascertained on a large scale with all the sludge produced in a town of 150,000 inhabitants over a period of 14 or 15 months, and, so far as he could judge, every sludge would be amenable, or could be made amenable, to the treatment described, no matter how large the town and the quantities to be dealt with might be; he had no doubt that the method could be applied to Birmingham sludge. The liquor which separated from the sludge in his concentration tanks was returned to and mixed with the raw sewage. It only amounted to a small fraction of the total sewage and was beneficial to it, as it contained iron and aluminium salts which had been dissolved out of the sludge by the acid. Although this paper only dealt with the preliminary concentration of sludge and not with his process of grease recovery, he could say, in reply to the question raised, that the de-greased residue produced by his complete process when mixed with suitable other compounds had yielded remarkably good results to farmers. His calculations with regard to the cost of drainage of sludge on cinder beds or similar arrangements were based on the ordinary prices accepted by contractors for excavation work. Considering that it was necessary to dry sludge in such a manner as to prevent over-heating, he considered the result which he obtained, i.e., of evaporating 6 lb. of water with 1 lb. of coal, as highly satisfactory; if the operation were to be carried out under pressure, the temperature would be higher and grease would be destroyed; there would be also great mechanical difficulties, which would render the apparatus too complicated and expensive. The points raised with regard to filter-pressing had been fully dealt with in his paper "Notes on Sewage Sludge and its Disposal" (this J., Jan. 15, 1912).

In that paper he showed, on figures published by Mr. A. B. Ogden, the then Manager of the Main Sewage Works of the Manchester Corporation at Davyhulme, that the cake which he (Mr. Ogden) assumed contained 50% of solid matter and cost 2s. per ton to produce, could not possibly have contained more than 27.3% of actual sludge, and that the cost of filter-pressing would have been nearer 4s. than 2s. per ton of cake, and probably, taking all matters into consideration, 7s. 4d. per ton of actual dry sludge. He wished again to point out that for purpose of calculations of cost in concentrating or removing sludge by different methods, it was absolutely necessary to reduce the figures to dry actual sludge. There was no perceptible increase in the volume of the sludge caused by treatment with acid although an evolution of gas took place. When mixed with acid, the sludge could remain in the tanks for five or six days without becoming septic, but he could not say whether if spread on the land afterwards it would retain that property. The Oldham sludge was obtained by ordinary sedimentation, and occasionally also a small quantity of aluminio-feric was added to it. There was no doubt that sodium bisulphate (nitrecake) could be used instead of sulphuric acid if, including carriage, etc., the available acid in it could be obtained as cheaply as commercial vitriol.

## New York Section.

Meeting held at Rumford Hall, Chemists' Building, on Friday, April 23rd, 1915.

MR. G. W. THOMPSON IN THE CHAIR.

### PAINT VEHICLES AS PROTECTIVE AGENTS AGAINST CORROSION.

BY MAXIMILIAN TOCH.

A careful search of the literature of the past twenty years has failed to reveal anything like a systematic investigation of the relative value of different vehicles used in the manufacture of paints for structural steel and the prevention of corrosion. There are a few isolated cases in which boiled linseed oil,\* Kauri linseed oil varnish† and spar varnish as protective coatings on structural steel were studied. For many years past much has been written and many investigations have been made on the protective quality of the pigments, but no one has apparently made any study of the vehicles.

It is quite obvious that without a vehicle a pigment is useless, and I know of no instance where a pigment could be used alone, with perhaps the single exception of Portland cement, if that may be classed as a pigment, and, then, Portland cement would be useless unless water is used as a vehicle.

To examine the comparative values of the various vehicles, exposure tests were made in 1913, in which fifty-two steel plates were carefully freed from grease by washing with benzol, dried, sanded, and rubbed clean with pumice, and then coated with the vehicle. Fifty-two paint vehicles or protective vehicles were examined, many of which, of course, are seldom, if ever, used alone, and some of which are failures a short time after they are put on. However, I wished to make the investigation

\* C. Von Kreyzig, *Farben-Zeit.*, 17, 1768-8. J. N. Friend, Carnegie Scholarship Report, Iron and Steel Inst., May, 1913, 1-3.  
† Address of Prof. A. H. Saltin before American Society of Civil Engineers, Nov. 4, 1906 (*Engineering News*, July 28, 1906).

## Inspection Report on Steel Plates exposed December 8th, 1913.

Pl. No.	Coating.	April 14th, 1914.	December, 1914.	April, 1915.	Value.
1	Raw linseed oil .....	Completely rusted	Completely corroded	Completely corroded	1
2	Raw linseed oil plus 10% linoleate drier .....	About $\frac{1}{2}$ rusted	Badly rusted along edges; partly protected in middle	Completely corroded	1B
3	Raw linseed oil plus 10% Japan drier .....	Slight rusting	(1) Slight rust along edges; fairly protected over rest of film (2) Badly rusted along edges; partly protected in middle	Completely corroded	1B
4	Raw linseed oil plus 10% Japan drier .....	Almost completely rusted	Almost completely corroded	Completely corroded	1
5	Japan drier .....	Completely rusted	Completely corroded	Completely corroded	1
6	Menhaden oil plus 10% Japan drier .....	Completely rusted	Completely corroded	Completely corroded	1
7	Menhaden oil .....	Almost completely rusted	Completely corroded	Completely corroded	1
8	Soya bean oil .....	Completely rusted	Completely corroded	Completely corroded	1
9	Soya bean oil plus 10% light drier .....	About $\frac{1}{2}$ rusted	Much corroded on upper half, free from rust on lower half	Completely corroded	1B
10	Soya bean oil plus 10% linoleate drier .....	About $\frac{1}{2}$ rusted	Much corroded on upper half, free from rust on lower half	Completely corroded	1B
11	$\frac{1}{2}$ raw oil, $\frac{1}{2}$ menhaden oil plus 10% linoleate drier .....	About $\frac{1}{2}$ rusted	Much corroded on upper half, free from rust on lower half	Completely corroded	1B
12	$\frac{1}{2}$ raw oil, $\frac{1}{2}$ soya bean oil plus 10% linoleate drier .....	About $\frac{1}{2}$ rusted	Badly rusted $\frac{1}{2}$ way down; fairly good on lower fourth of plate	Completely corroded	1B
13	Perilla oil .....	Completely rusted	Completely corroded	Completely corroded	1
14	Perilla oil plus 10% Japan drier .....	Almost completely rusted	Completely corroded	Completely corroded	1
15	Petrolatum .....	Much dirt, slight rusting	Collected much dirt; but well protected from rust	Still very good	4
16	Spar varnish .....	No rusting	Slight rusting	Unchanged; very good	5
17	Turpentine substitute .....	Completely rusted	All corroded	All corroded	1
18	Anhydrous pine oil .....	Completely rusted	All corroded	All corroded	1
19	Benzine .....	Completely rusted	All corroded	All corroded	1
20	Turpentine .....	Completely rusted	All corroded	All corroded	1
21	Paraffin oil .....	Completely rusted	All corroded	All corroded	1
22	Boiled linseed oil and turpentine substitute thinner .....	No rusting	No rusting	Unchanged; very good	5
23	Boiled linseed oil and turpentine substitute thinner .....	No rusting	Very slight rusting	Unchanged; very good	5
24	Peroxylin lacquer .....	Completely rusted	All corroded	All corroded	1
25	Celluloid solution .....	Completely rusted	All corroded	All corroded	1
26	No. 38 China wood oil varnish .....	Slight rusting	Rusted appreciably under whole film	Rather badly corroded	2
27	No. 5 copal, China wood oil varnish .....	No rusting	No rusting	Rather badly rusted	2
28	No. 6 copal, wood oil varnish .....	Slight rusting	Rusted much over whole plate	All rusted	2
29	No. 39 China wood oil varnish .....	Slight rusting	Rusted under whole film to small extent	All rusted	2
30	Steam cylinder oil .....	Completely rusted	Completely corroded	All rusted	1
31	Gear case oil .....	Much dirt; slight rusting	Collected very much dirt; shows but little rusting	Still protected	4
32	Blown linseed oil plus turpentine .....	Slight rusting	(1) Slight rusting in few spots (2) Badly rusted all over	—	2
33	Kerosene oil .....	Completely rusted	Completely corroded	Completely corroded	1
34	Raw oil plus 2% cobalt drier .....	About $\frac{1}{2}$ rusted	Rusted badly $\frac{1}{2}$ way down	Completely corroded	1B
35	Raw China wood oil .....	About $\frac{1}{2}$ rusted	Almost completely corroded	Completely corroded	1
36	$\frac{1}{2}$ raw oil, $\frac{1}{2}$ raw China wood oil .....	About $\frac{1}{2}$ rusted	Badly corroded $\frac{1}{2}$ way down	Completely corroded	1
37	Blown linseed oil, turpentine substitute, 10% linoleate drier .....	Slight rusting	Partly rusted in quite a few isolated spots	All corroded	2
38	Raw oil, 10% paraffin oil, 10% Japan drier .....	About $\frac{1}{2}$ rusted	Badly rusted $\frac{1}{2}$ way down	All corroded	1B
39	China wood oil, rosin varnish .....	No rusting	No rusting; very good	Badly corroded	3
40	Heavy boiled linseed oil, $\frac{1}{2}$ raw oil .....	No rusting	No rusting; very good	Badly corroded	3
41	$\frac{1}{2}$ spar varnish, $\frac{1}{2}$ stand oil .....	No rusting	No rusting; very good	Still good	5
42	80% raw oil, 15% spar varnish, 5% linoleate drier .....	No rusting	Rusted a little along upper edge and in several other spots	Badly rusted	3
43	$\frac{1}{2}$ raw oil, $\frac{1}{2}$ No. 6 copal wood oil varnish, 5% Japan drier .....	No rusting	No rusting; very good	Badly rusted	3
44	$\frac{1}{2}$ heavy boiled linseed oil, $\frac{1}{2}$ menhaden oil, 5% Japan drier .....	No rusting	Very slight rusting	Badly rusted	3
45	$\frac{1}{2}$ heavy boiled linseed oil, $\frac{1}{2}$ soya bean oil, 5% Japan drier .....	No rusting	No rusting; very good	Badly rusted	3
46	Kettle boiled oil .....	No rusting	Practically no rusting	Still very good	5
47	Pale refined varnish oil .....	Completely rusted	Completely corroded	All corroded	1
48	Pale refined linseed oil .....	Completely rusted	Completely corroded	All corroded	1
49	80% wood oil, 10% No. 38 China wood oil varnish, 10% Japan drier .....	No rusting	Practically no rusting	Corroded	3
50	90% raw oil, 10% paraffin oil, 1% cobalt drier .....	No rusting	Somewhat rusted on top; perfectly good at bottom	Still good	5
51	95% spar varnish, 5% petrolatum in turpentine substitute .....	No rusting	Slight rusting underneath film along edges	Still good	5
52	China wood oil, heated with Tox-tungate and thinned with benzine .....	No rusting	No rusting at all; very good	Still very good	5

as complete as possible, and for this purpose I selected the same quality of steel, known as cutlery steel, which rusts very rapidly, and which I have been using for many years for exposure tests.

Those plates which have shown no rusting in the year and five months that they have been exposed must be eliminated, since they were coated with the paraffin or machinery oil compounds, and an engineer would not coat steel with paraffin compounds, because the cleaning before the application of any good paint would have to be very carefully carried out, as no protective paint would hold on steel coated with a paraffin base unless it were entirely removed. Then the paraffin, or non-drying oils, all collect a great deal of dirt, and would have to be entirely removed before any paint could be applied.

Plate No. 41 showed excellent results, and a material of this kind would not be so very expensive where engineers demand that steel be coated with a clear liquid in the shop so that the steel may be inspected in the field. This was composed of half spar varnish and half "Stand oil" which is practically a polymerized linseed oil. Linseed oil when heated to 550° with a drier like Japaner's Brown or borate of manganese will produce a very thick viscous liquid, which is largely used as a patent leather finish. This can be reduced with 50% of thinner and still have the fluidity or viscosity of raw linseed oil, and is therefore inexpensive.

Plate No. 50 was coated with a material containing 10% of paraffin oil, which might be classed as an adulterated linseed oil, and while it showed up very well, it could not be recommended because on an exposed structure, like a bridge, a coat of good protective paint would not adhere very thoroughly.

Plate No. 52 was treated with raw China wood oil which had been heated sufficiently to take 10% of a tungate drier, and then thinned with 15% of benzine. This made a material which is hardly more expensive than good boiled linseed oil, and left a most excellent surface for re-painting. In fact, this has proved itself the equal of plates No. 22 and No. 23, and also offers a better surface for re-painting.

Plate No. 46 was coated with kettle-boiled linseed oil, and is very good, but this material might be regarded by some engineers as too expensive for application, as it took all day to make this oil. A carefully selected linseed oil was chosen to start with, to which was added 5% of litharge and no other drier. This oil dried very slowly, but produced a good flexible film which lasted. This must not be confounded with the average boiled linseed oil of commerce.

The various coatings used in these exposure tests (see table on page 593) have been divided according to their protective value into five classes:—

1 and 1B. This class includes those vehicles which have little or no value for the prevention of rusting. It contains:—

(A) The raw and refined drying and semi-drying vegetable oils. (Plates Nos. 1, 7, 8, 13, 35, 36, 47, 48).

(B) The same oils to which 10% of drier has been added (Plates Nos. 2, 3, 4, 6, 9, 10, 11, 12, 14, 34).

(C) The more or less volatile paint thinners (Plates Nos. 17, 18, 19, 20, 33).

(D) Solutions of celluloid and pyroxylin (Plates Nos. 24, 25).

(E) Paraffin oils liquid at room temperature (Plates Nos. 21, 30).

2. Here have been included those vehicles which showed some degree of protection, though not very much at best.

(A) Wood-oil-varnishes containing a certain percentage of rosin (Plates Nos. 26, 29).

(B) Copal-wood-oil varnishes (Plates Nos. 27, 28).

(C) Varnishes made from linseed oil which have been thickened and oxidized by blowing with air, oxygen, or ozonized air (Plates Nos. 32, 37).

This compared with the results obtained below with cooked-oil varnishes proves conclusively that the film yielded by a blown oil is not nearly as waterproof and resistant to severe weather conditions as that formed by a boiled or polymerized oil.

3. This class includes the varnishes or varnish mixtures which protected the steel very nicely as long as weather conditions were not severe and temperature changes not very rapid and pronounced. (Plates Nos. 39, 40, 42, 43, 44, 45, 49).

4. To this class belong the semi-solid and solid paraffin oils. These show a very high degree of protection from rusting. (Plates Nos. 15, 31.)

5. Here we have set down the varnishes and vehicles which afford a high degree of protection against corrosion. To be put in this class a material must be extremely water-proof, it must dry with a film which is very elastic and yet tough in order to be able to withstand "weathering." A film which cannot remain intact against condensed moisture, snow, and ice and despite comparatively wide and sometimes rapid changes in temperature (as between day and night even in rather warm climates) will of necessity afford very little protection for the steel to which it is applied.

As the table shows this class comprises:—

(A) Spar varnish (Plate No. 16).

(B) Varnishes made from linseed oil or China wood oil, which has been thickened by a heat process (Plates Nos. 22, 23, 52).

(C) Open kettle-boiled oil (Plate No. 46).

In plate No. 50 we find a rather anomalous case. It seems that raw linseed oil which has been dried with a small percentage of a liquid paraffin oil proved to be an excellent coating for rust prevention.

The addition of any paraffin or non-drying oil, even in such a small quantity as is shown in Plate No. 50, is dangerous in case repainting becomes necessary. Although I cannot yet state definitely whether linseed oil and paraffin oil dissolve in each other, my idea at present is that, although they apparently make a clear solution, separation takes place. I have made several experiments, and find that a film of linseed oil which contains paraffin oil in some quantity, when apparently dry shows minute globules of paraffin oil in liquid form when the film is heated above 100° C. A film of linseed oil containing 10% of paraffin oil after it is six months old can be shown to contain uncombined paraffin oil by extraction with naphtha. These experiments prove conclusively that it is dangerous to mix a paraffin oil with linseed oil for any purpose, excepting where it is not necessary, or not the intention, to repaint subsequently.

#### DISCUSSION.

THE CHAIRMAN asked whether the oil which gave such good results was a kettle-boiled oil or a commercial boiled oil, and what was its gravity?

Mr. TOCH replied it was a kettle-boiled oil which he had made himself; he did not know its exact gravity, but was sure it was over 0.980.

Mr. R. H. GAINES confirmed Mr. Toch's statement that heavy paraffin oil protected steel against rust but collected dirt and cinders in the meantime.

Mr. F. S. LOW asked whether Mr. Toch had any data on the viscosity of these oils previous to their being applied, for it appeared to him that the value of the oil depended upon the thickness of the coating, and that was probably why the kettle-boiled oil (panel No. 46), and the boiled China wood

oil with the tungate drier (panel No. 52) produced such good results.

Mr. TOCH agreed that the thickness of the coating was in direct ratio to its protective quality, provided it had a protective quality to start with, and assuming, of course, that it was a drying oil. He had not made any measurements of the thickness of the various films, but those that gave good results showed an appreciable thickness of film.

Prof. A. H. SABIN said that a shop coat of linseed oil for bridge work had not entirely gone out of use. There were several different reasons for applying it. The structural metal, as received from the shop, was covered with mill scale. Sooner or later it would come off, carrying with it any paint that had been applied. The shop coat of linseed oil was applied, and the bridge erected; the mill scale was allowed to rust off, and then the bridge was thoroughly cleaned and painted. On another railroad a shop coat of linseed oil was applied, the bridge erected and then painted by the company's painters. Others held that a shop coat of linseed oil enabled the inspector to see whether the rivetting, etc., had been properly done, better than when the work had been painted. The question was whether it was possible to get a vehicle that would work more easily than linseed oil and could be sold at a suitable price.

Mr. STEVENSON said that in the erection of steel the shop coat was almost always torn off in the rough handling it received. It cost more to apply a coat of paint in the shop than it did in the field. Linseed oil gave protection for four or five months, and that was as long as was necessary, and had given very good results except in one or two cases.

(see this Journal, 1915, 62—65). I pointed out that when considering the indications given by such an instrument as the Cloth Oil Tester it should be remembered that one was dealing with the oil in very intimate contact with cotton wool, and that probably the action between the oil and the cotton had something to do with the result. I suggested experiments in which the oil should be spread on some inert substance such as glass wool.

Subsequently Mr. Miller, my assistant, pointed out that experiments had already been made on these lines, and looking back over the laboratory note-books we found that some work had been done in 1895—96. These experiments are now described, together with some experiments made since the meeting already referred to; and they do not bear out the opinion I then expressed. At any rate the heating proceeds rather more rapidly when slag or glass wool is used instead of cotton wool (see Tables 1 and 2).

Tables 3 and 4 deal with oils tested in the usual way in the Oil Tester—that is, spread on cotton wool—and are given to show that the iodine value alone does not give reliable indication as to the behaviour of an oil as regards liability to induce spontaneous heating when spread on fibre.

Table 3 gives the results of experiments on hardened cottonseed oil (samples kindly given by Dr. Armstrong, of Messrs. Joseph Crosfield & Sons, Ltd., Warrington), and are of some interest apart from the question of iodine value, etc.

It will be noted in the case of the "semi-hardened" oil that the free fatty acids "cracked out" and dried (containing any unsaponifiable matter present) give a decided rise of temperature in the Oil Tester, whilst the oil does not; the point of interest is that the iodine value of the free fatty acids is not substantially higher than that of the neutral oil. It will be noted further that the free fatty acids from the cottonseed oil "hardened nearly to saturation" (as described by Dr. Armstrong) do not give any rise of temperature. The iodine value of these was found to be 2.5 (determined by Dr. Ingle). The neutral oil itself was not tested in the Oil Tester, it being presumed to give no rise in temperature.

In Table 4 we have two oleines of similar composition as regards unsaponifiable matter and free fatty acids, and similar iodine value, showing a decided difference in behaviour in the Oil Tester—A (iodine value 81.8) giving no rise, B (iodine value 87.5) showing a very rapid rise, being evidently a distinctly dangerous oil as regards liability to induce spontaneous heating when spread on fibre.

## Yorkshire Section.

Meeting held at Leeds, on Monday, April 19th, 1915.

MR. F. W. RICHARDSON IN THE CHAIR.

### NOTE ON THE BEHAVIOUR OF SOME OILS AND FATTY ACIDS IN MACKEY'S CLOTH OIL TESTER.

BY WILLIAM MCD. MACKEY.

In the discussion on the paper by Hyland and Lloyd, "The Oxidation of Oils and Fatty Acids"

TABLE 1.

		1h. 0m.	1h. 15m.	1h. 30m.	2h. 0m.	Maximum	h. m.
Same sample	Olive oil on cotton wool	95°C.	—	99°C.	103°C.	105°C. in	3 0
	" " wool	92.2°C.	98°C.	101.5°C.	105°C.	109.8°C. "	4 45
	" " slag wool	101°C.	104°C.	105.8°C.	109°C.	111°C. "	3 50
Same sample	" " cotton wool	96.5°C.	98.5°C.	100°C.	104°C.	Experiment stopped	
	" " glass wool	102°C.	108°C.	115.5°C.	—	129°C. in	1 55

TABLE 2.

	1h. 0m.	hrs. min.		
Raw linseed oil on cotton wool, typical of a number of experiments	110° C.	200° C. in 1 11	Thermometer withdrawn	
Raw linseed oil on slag wool		200° C. " 0 43	" "	
Boiled linseed oil on slag wool		200° C. " 0 28	" "	
Same Sample	Cottonseed oil on cotton wool	110° C.	200° C. " 1 10	Temperature began to fall after 1 hour 15 min.
	" " slag wool	173° C.	191° C. " 1 10	

NOTE.—In the case of the slag wool 32 grms. was used and in that of the glass wool 23 grms.

TABLE 3.

	1h. 0m.	1h. 15m.	1h. 30m.	2h. 0m.	
Semi-hardened cottonseed oil on cotton wool, iodine value 77.6	95.2° C.	96° C.	96.8° C.	97.2	Experiment stopped
Fatty acids from above sample on cotton wool, iodine value 79.4	111.3° C.	118° C.	—	—	202° C. in 1 hour 24 min. Thermometer withdrawn
Fatty acids from hardened cottonseed oil on cotton wool ("hardened nearly to saturation")	93.5° C.	95.3° C.	96° C.	96° C.	Experiment stopped

TABLE 4.

	1h. 0m.	1h. 15m.	1h. 30m.	2h. 0m.	2h. 15m.	
Olefin "A" on cotton wool, iodine value 81.8	95° C.	96° C.	96.8° C.	97.6° C.	98.8	Experiment stopped
Olefin "B" on cotton wool (similar to above as regards unsaponifiable and free fatty acids) iodine value 87.5	107° C.	222° C.	Thermometer withdrawn			

## DISCUSSION.

Mr. F. W. RICHARDSON said that the iodine value of an oil was apparently not so reliable a factor as he had thought in regard to oxidation. In his opinion it was a question whether oxidation under ordinary conditions was comparable with oil tested in the Cloth Oil Tester. His experience was that, all things being equal, the iodine value indicated fairly well the increase of viscosity and the oxidation changes which would take place when an oil was exposed at the ordinary temperature.

Dr. H. INGLE said that Mr. Mackey's results proved that the heating of oils in the Tester, and presumably on the wool, etc., depended upon the surface of oil exposed to the air and not upon any action of the oil upon the fibre. It would be interesting to compare these surfaces of cottonwool, wool, and glass wool, but it was clear that the animal and vegetable fibres might absorb oil into their mass and so reduce the surface layer of the oil exposed. That would explain why in some cases a greater rise in temperature was recorded upon glass wool than on cotton wool, since glass wool was impervious to oil.

The experiments with boiled linseed oil showed the effect of a drier increasing the rate at which the oil would absorb oxygen. The rise in temperature in all cases must depend upon the rate of the oxidation, and the experiment with boiled linseed oil illustrated that point admirably. The iodine value (which, taking all in all, was one of the best tests) only gave the amount of oxygen that might be absorbed, not the rate at which it would happen. He (the speaker) had shown that the acids of linseed oil took over twenty months to dry (this Journal, 1913, p. 639), while the glycerides would dry in a few days, although the iodine value of the acids was higher than that of the glyceride. The same applied to the ethyl esters, as had been shown by himself and also by Dr. Lloyd. However, the actual amount of oxygen absorbed by the acids and ethyl esters was only half that absorbed by the glycerides.

It was the rate of oxidation which determined the rise in temperature, for even if the actual oxygen absorption, measured by direct weighing or by the iodine value, were great, if the rate of absorption were slow, the heat generated was dissipated and no great rise in temperature resulted.

Reverting to the differences in the results which Mr. Mackey had obtained with the free acids and their glycerides, great difficulty was found in explaining this. One suggestion he (the speaker) offered was that decomposition of the peroxide of an acid (at the temperature of the tester) by the formation of alcoholic or aldehydic groups in the

presence of the free acid might give lactones, while these could not be formed from the glycerides. Whether the formation of lactones would give rise to sufficient heat to cause the marked increase in temperature noted by Mr. Mackey was doubtful. Water would aid in the decomposition of the peroxides and the formation of hydroxyl groups, and it had been shown by both Mackey and Lloyd (this Journal, 1915, p. 62) that moisture increased the heat developed in the tester.

He did not think that any appreciable polymerisation of ordinary oils took place below 500° F.

Mr. W. RUSHBY said that when slag wool was used in the Tester, no moisture was present, but if wool or cotton wool were used moisture would be present during the testing, because moisture was naturally present in wool up to 15% and cotton up to 8%.

Mr. RICHARDSON said that his experiments bore out Dr. Ingle's statement. If a level surface were used, the oxidation was proportional to the area of the surface. That was the reason slag wool gave higher results than either wool or cotton wool.

Dr. L. L. LLOYD said that he had carried out some experiments similar to those of Mr. Mackey's. The materials were all thoroughly scoured with benzene, water, and a little potash soap made from stearic acid, then washed with water and air-dried. The method of mixing with the oils and the compactness of the oiled material were approximately similar. The results are given in the following table:—

Oil.	Fibre material.	Max. temp.	Time for max. temp.	Decrease in iodine value.
Olive I.v. 86.8	Glass wool	130° C.	5h. 45m.	37.2
	Cotton "	130° C.	5 40	36.0
	Wool "	135° C.	6 15	43.6
Cotton seed I.v. 108.7	Silk	185° C.	6 30	35.1
	Glass wool	209° C.	1 15	36.8
	Cotton "	210° C.	1 15	35.2
Cotton seed hydrogenated I.v. 35.0	Wool "	203° C.	1 20	46.3
	Silk	190° C.	1 15	37.1
	Glass wool	96.3° C.	5 30	1.8
Olefin 70% I.v. 83.2	Cotton "	95.8° C.	5 30	1.8
	Wool "	97.3° C.	5 30	1.6
	Silk	96.1° C.	5 30	7.9
	Glass wool	98.2° C.	5 30	6.8
	Cotton "	96.5° C.	5 30	7.3
	Wool "	97.3° C.	5 30	6.0
	Silk	96.8° C.	5 30	

The product extracted from the wool containing the olive oil, after heating in the Mackey Tester, was very dark and was found to contain sulphur which was no doubt obtained by the decomposition

of the wool. Similarly the cottonseed oil product extracted from the wool also contained sulphur.

From the above figures it appeared that the nature of the material upon which the oil was spread was of little consequence as regards oxidation. It was, however, different where oxidisable oils were exposed, when heating effects took place, because wool, silk, and inert materials did not fire, but cotton fired and burned fairly easily, the cotton charring at about  $180^{\circ}$  upwards.

Olive oil exposed to the oxidising action of air, when treated in the Mackey Tester, did not show any appreciable heating effect. The oil, however, became oxidised, and after extraction had decreased in iodine value; the oil was also somewhat thicker. If, however, the material were left in the Mackey Tester for about 4 to 5 hours, then the heat produced by oxidation or other causes became apparent and the rise in temperature took place quickly. If olive oil were exposed to the air at ordinary temperature, the absorption of oxygen took place so slowly, that after three years' exposure, in a thin film, the iodine value was reduced only by about 4 to 5%. That the iodine value did not govern the liability to oxidation and firing was proved from the following experiments in the Mackey Tester. Fatty acids of iodine value 77.8 registered  $99.5^{\circ}$  C. after five hours' treatment in the Tester, ethyl oleate (pure) of iodine value 75.6 registered  $213^{\circ}$  C. in five and a half hours, the ethyl oleate rising at first extremely slowly and finally very rapidly.

Mr. J. HYLAND, referring to the paper of Hyland and Lloyd, said that by oxidising oils on quartz at  $50^{\circ}$  C. until a maximum gain in weight was reached, a decrease in iodine value resulted very similar to that found after the oil had been through the Mackey Tester. Ethyl oleate gave a decrease in the tube of 31.4 and in the Mackey Tester 30.8. Also those oils with which a theoretical increase in weight was not reached on account of the decomposition of the oxidation products, were found to be more dangerous when tested in the Mackey Tester; and this, coupled with the fact that moist air was shown to aid in the decomposition of the oxidised oils, and particularly of the fatty acids, probably helped to explain why moist air should aid in the firing of the oils when on the fibre. That the nature of the fibre is of some importance from a firing standpoint was shown from the treatment of "Black Oil Cake," obtained in the Heavy Woollen District. Cake containing cotton fibre could not be stored without risk of firing, whereas cake free from cotton fibre might be stored for a considerable period.

## Communications.

### A SIMPLE INSTRUMENT FOR THE DETERMINATION OF VISCOSITY.

BY ALAN SPEEDY.

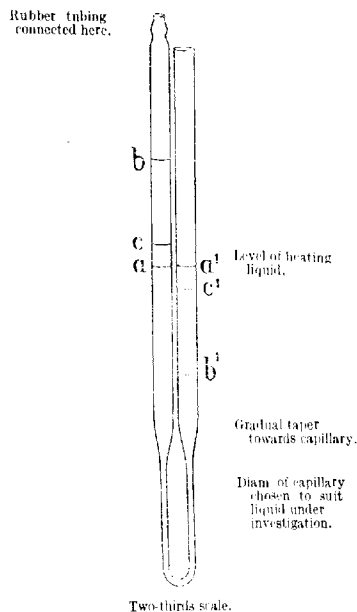
In the determination of viscosity by the aid of viscometers of the Ostwald type, it is customary to immerse the whole apparatus in a medium of high boiling point. The use of a fairly large quantity of some oil or wax transparent at the temperature of observation is thereby necessitated.

In the simple instrument described below, all difficulty in reading the marks is avoided by placing them above the surface of the liquid, which, therefore, need not be transparent and of which only a moderate quantity is required.

The instrument consists of a piece of glass

tubing drawn out to form a capillary and bent into U shape, as shown in the figure.

The liquid of which the viscosity is to be determined is filtered into the viscometer, which is clamped vertically in the bath so that the level



marks,  $aa'$ , are just above the surface of the heating liquid. The bath is now heated to the required temperature and after a few minutes the levels,  $aa'$ , are carefully adjusted. By means of a piece of rubber tubing the liquid is then slowly sucked up the left limb of the tube until it passes the level,  $b$ . It is then allowed to descend. The time taken to fall from  $b$  to  $c$  is recorded on a stop watch, and the experiment repeated as a check.

Part of the liquid under observation is above the surface of the heating liquid and is, therefore, at a slightly lower temperature, but as the level falls, this part of the liquid regains its former temperature. The error due to unequal viscosity in different regions arising in this way is negligible. It is only the viscosity of the liquid in the capillary tube that counts. The thinness of the wall of the capillary tube ensures that the liquid passing through the tube is at the temperature of the bath. It should be observed that the column of liquid,  $bc$ , never reaches the capillary. The level,  $b'$ , is well above the point where the constriction in the right limb of the tube begins. The time of flow is taken from  $b$  to  $c$  only, because on approaching  $a$  the motion becomes slow and uncertain.

The constant of the instrument can be obtained by calibrating it with pure phenol (see A. E. Dunstan, this J., 1912, 34, 1063) or sulphuric acid (see Proc. Chem. Soc., 1911, Vol. 80).

The instrument may conveniently be strapped to a thermometer and suspended in the heating liquid, which is contained in a boiling tube. The cheapness of the apparatus renders it possible to choose from a range of tubes one with a capillary of diameter best suited to the viscosity of the liquid which is to be investigated. The best results are obtained if the time of flow is about 1-2 mins. at the temperature of observation.

As an illustration of the degree of accuracy that may be expected from this form of viscometer, the examples contained in the table below are given.

Six oils, of which five were kindly supplied by Messrs. Ragosine and Co., were tested for viscosity in the instrument just described, and also, for the sake of comparison, in two other forms of viscometer, of which one is the totally immersed type,\* and the other the well-known Redwood form.

Knecht's process of titration with titanous chloride (J. Soc. Dyers and Col., 1905, 21, 3—6; see this J., 1905, 154) to determine the amount of fading and washing out, etc., of a series of azo dyes, but after numerous trials it was abandoned. It was thought that probably the titanous chloride is so strong a reducing agent that under the conditions of the titration it to some extent decomposes the water of the solution or the carbon dioxide atmosphere in which the reduction is carried out.

Sample.	Nominal sp. gr.	T°	n Dunstan.	n New type.	Secs. Redwood.	Closed dash pt.°F.
Genuine refined pale East Indian rape oil .....	0.910	60° F. 70° F. 140° F. 200° F.	— 0.7680 0.1864 0.0815	— 0.7669 0.1872 0.0818	535 312 98 55	
American pale oil A. ....	0.900	70° F. 140° F. 200° F. 250° F.	0.7600 0.1230 0.0497 0.0283	0.7592 0.1224 0.0495 0.0283	348 69 41 34	300
American pale oil B. ....	0.908	70° F. 140° F. 200° F. 250° F.	— 0.1580 0.0583 0.0328	— 0.1575 0.0578 0.0327	400 80 45 36	400
Russian pale oil .....	0.908	70° F. 140° F. 200° F. 250° F.	— 0.2624 0.0853 0.0421	— 0.2611 0.0859 0.0423	1235 131 53 40	384
Neptune brand lubricating oil ....	0.850	150° C. 180° C. 200° C.	0.0718 0.0450 0.0348	0.0714 0.0452 0.0355		
Shale oil distillate .....	0.840 to 0.860	40° C. 80° C. 100° C.	0.0316 0.0145 0.0114	0.0319 0.0149 0.0117		

The readings of the first two instruments agree within the limits of experimental error. At 140° F. it will be seen that Redwood seconds may be obtained approximately by multiplying these readings by 500.

It is believed that the extreme simplicity of the instrument, and the increased accuracy due to choice of a suitable capillary, as well as its convenience in use, may recommend it to all who have measurements of viscosity to make.

#### THE USE OF KNECHT'S PROCESS FOR DETERMINING THE FASTNESS OF DYES TO LIGHT AND OTHER AGENCIES.

BY ARABINDA SIRKER.

In a previous investigation carried out in this laboratory (Watson, Sirker, and Dutta, this J., 1911, 30, 6-9) an attempt was made to use

\* A. E. Dunstan, *ibid.*

Further experiments proved that this surmise was not correct, and that the difficulties previously experienced were due to the presence of air in the apparatus. For the titration of dyes which are not readily reduced it was necessary to exclude completely every trace of air from the apparatus. When this was done Knecht's process gave quite satisfactory results. It has been found that dyes can be estimated on wool as well as on cotton.

*Preparation of dyed samples.*—Well-washed wool was dyed in each case in a bath containing 2% dyestuff and 2% sulphuric acid (on the weight of wool) by boiling for one hour. In each titration, about 100 c.c. of water and 15–20 c.c. of concentrated hydrochloric acid were added to the material to be examined, excess of titanous chloride was added, and the excess was determined by means of standard ferric alum solution. Every precaution was taken to prevent access of air.

Dye.	Mol. wt.	Theoretical value of 0.04 grm. of each dyestuff expressed in c.c. of titanous chloride (1 c.c. 0.00166 grm. Fe).	Titanous chloride solution required by the bath after dyeing c.c.	Titanous chloride solution required by the dyed wool (2 grms.) c.c.
1. Benzene-azo-phenol .....	198	27.29	8.07	19.22
2. Benzene-azo-resorcinol .....	216	25.01	2.90	22.11
3. Sulphobenzene-azo-phenol .....	278	19.43	2.76	4.17
4. Benzene-azo-phenol-sulphonie acid .....	278	19.43	0.70	18.73
5. Benzene-azo-salicylic acid .....	242	22.23	1.20	21.03
6. Bromobenzene-azo-phenol .....	277	19.50	1.01	18.49
7. Nitrobenzene-azo-phenol .....	243	35.57	14.49	39.28
8. Benzene azo-dibromoniline .....	355	15.22	3.59	11.46
9. Amino-azo-toluene .....	225	24.91	0.97	23.04
10. Amino-azo-benzene .....	197	27.29	10.06	17.28
11. Chrysoline .....	265	18.95	15.72	3.25
12. Orange II .....	329	16.46	0.970	15.48

The figures in the third column were obtained by calculation, after it had been ascertained that the titanous chloride value of 0.04 grm. of dyestuff for four or five substances differed only by 1 to 1.5% from the theoretical values.

For testing the fastness to light, the samples were exposed at Dacca, in February and March, 1910. The standard samples for these dyeings could not be obtained, therefore the actual quantities of dyestuff which they contained before exposure could not be ascertained. The intensities after exposure were calculated on the assumption that the dyeings originally contained the same amounts of dyestuffs as those dyed under the same conditions and used for the soap, alkali, and acid tests.

The following table shows the results of the tests of fastness to light of various dyes on wool:—

No. Dyeing.	Intensity of shade after 22 days' exposure, expressed as percentage of original intensity.	Percentage of fading in 22 days.	Intensity of shade after 59 days' exposure, expressed as percentage of original intensity.	Intensity of shade after 59 days' exposure, calculated by assuming the intensity after the 22nd day as the original intensity.
1. Chrysoidine .....	93.5 (?)	—	75.0 (?)	—
2. Benzene-azo-resorcinol .....	88.0 (?)	—	74.0 (?)	—
3. Benzene-azo-phenol .....	87.0	13.0	81.0	93.1
4. Benzene-azo-sulphilic acid .....	83.6	16.4	72.2	87.5
5. Amino-azo-benzene .....	82.0	18.0	62.0	75.6
6. Benzene-azo-phenol- <i>o</i> -sulphonic acid .....	75.6	24.4	66.2	87.5
7. Sulphobenzene-azo-phenol .....	67.6	32.4	5.6	82.2
8. Nitrobenzene-azo-phenol .....	61.0	39.0	3.0	86.8
9. Bromobenzene-azo-phenol .....	56.6	43.4	3.0	68.0
10. Orange II .....	30.0	70.0	1.0	7.0
11. <i>o</i> -Amino-azo-toluene .....	27.5	72.5	2.1	44.0
12. Benzene-azo-dibromoniline .....	13.4	86.6	1.0	29.8

\* In cases 1 and 2 the dye could not be reduced completely on the fibre, even in presence of a large excess of titanous chloride.

**Fastness to soaping, alkali, acid, and light.** The dyed samples were steeped for 15 minutes at 60° C. in an aqueous solution of neutral soap, containing 15 grms. per litre, rinsed out, and titrated. For the alkali test, they were steeped for 10 minutes in a solution of sodium carbonate (10 grms. crystal carbonate per litre) at 60° C. For the acid test, they were steeped for an hour in a 10% acetic acid solution at 40° C.

Dye.	Shade after soaping.	Shade after washing with alkali.	Shade after washing with acid.
1. Amino-azo-benzene .....	53.9	80.1	12.0
2. Benzene-azo-dibromoniline .....	50.2	62.0	98.9
3. <i>o</i> -Amino-azo-toluene .....	47.5	41.0	34.0
4. Nitrobenzene-azo-phenol .....	42.8	60.0	99.1
5. Benzene-azo-resorcinol .....	31.0	25.0	93.0
6. Nitrobenzene-azo-phenol .....	21.6	12.0	74.0
7. Chrysoidine .....	17.3	9.5	29.1
8. Benzene-azo-phenol .....	16.6	20.1	62.0
9. Orange II .....	10.2	18.0	70.0
10. Sulphobenzene-azo-phenol .....	7.0	1.0	80.1
11. Benzene-azo-phenol- <i>o</i> -sulphonic acid .....	4.1	7.1	90.2
12. Benzene-azo-sulphilic acid .....	3.8	13.0	97.0

The results are expressed as percentage of original intensity.

In discussing these results, benzene-azo-phenol was taken as the standard.

**Fastness to light.** None of the groups under consideration had a good effect.

The original unexposed samples corresponding to those which were exposed to light were lost. Therefore the original intensity is not known with much certainty. This introduces a possible error in the fastness determination.

Attempts were made to establish a relation between the intensity and the rate of fading,

but no satisfactory conclusion could be drawn. It seems quite probable that such a relation might be traced if several consecutive estimations were made at close intervals (say every 2 or 3 days).

Watson, Sircar, and Dutta place benzene-azo-dibromo-aniline in Class IV. and benzene-azo-phenol in Class V., therefore in order that my results may coincide with those found by them, the dyeings between benzene-azo-phenol and benzene-azo-dibromo-aniline ought to be placed in (Classes V, V.—IV., and IV.

Benzene-azo-dibromo-aniline and amino-azo-benzene are the two remarkable exceptions: the introduction of bromine atoms has a worse effect than these authors found, and amino groups had not such a marked effect. The anomalous behaviour of benzene-azo-dibromo-aniline may be due partly

to the fact that in the previous investigation the wool did not take up sufficient dye from the bath.

**Fastness to soaping and alkali.**—In both cases, introduction of amino groups or bromine atoms has a very good effect, increasing the fastness of benzene-azo-phenol about thrice. The methyl group increases the fastness more than two-fold, and the hydroxyl group is also beneficial. Introduction of sulphonic and carboxylic groups decreases the fastness.

**Fastness to acid.**—Chrysoidine, with two amino groups, retained only 29% of the total dye on the fibre, and amino-azo-benzene, with one amino group, retained 12%. Introduction of amino groups had therefore a bad effect. The other groups increased the fastness more or less.

These results agree with those obtained by Watson, Sircar, and Dutta.

**Practical results.**—Considering the results of all the determinations of fastness to various agencies, it is found that only bromine atoms can be introduced without much bad effect. Amino groups decrease the fastness to acid, and hydroxyl and sulphonic groups decrease the fastness to alkali and soap. When the benzene nuclei are replaced by naphthalene, fastness to soap, alkali, and acid is decreased.

The experiments show that the washing out of dyes by soap and alkali depends largely on the presence of acidic groups in the dye, amino-azo-benzene being quite fast and benzene-azo-phenol-*o*-sulphonic acid being practically washed out. In this respect the results do not quite coincide with those of Watson, Sircar, and Dutta.

This view is supported by the fact that dyeings with amino-azo-benzene are very largely destroyed by acid.

In conclusion, I beg to offer my best thanks to Dr. E. R. Watson, Senior Professor of Chemistry, Dacca College, for his kind help and advice.

The Dacca College,  
East Bengal, India.



## Obituary.

### JOHN JACOB BERINGER.

J. J. Beringer, who had been a member of this Society since 1884, died at Camborne on March 27th, 1915. During his earlier career, he was assistant to Professor Huntington at King's College, Lecturer to the Miners' Association of Cornwall, and Public Analyst to the County of Cornwall. He held the position of Principal at the School of Mines, Camborne, from 1882 to the time of his death. Beringer was a recognised authority on all matters relating to the dressing and assaying of tin ores, especially of those of Cornwall, and his original investigations and other work exercised a great influence in the improvements effected in the industry during the last 30 years. He was the author of a standard text-book on assaying and of a considerable number of pamphlets and papers published in the journals of technical societies. His influence in mining and metallurgical teaching has been exerted throughout the world, as his students were drafted to

the principal centres of tin mining and of most other branches of metalliferous mining.

### ARTHUR HERBERT CHURCH.

The death occurred at Shelsley, Kew Gardens, on May 31st, of Sir Arthur H. Church, Professor of Chemistry at the Royal Academy. He was born on June 2nd, 1834, and was educated at King's College and the Royal College of Chemistry and at Lincoln College, Oxford. In 1879 he was appointed Professor of Chemistry in the Royal Academy of Arts, and the following year became Lecturer on Organic Chemistry at Coopers Hill College.

Sir Arthur Church was a leading authority in the chemistry of painting, and gave valuable assistance in the work of preserving the paintings in the Houses of Parliament. He discovered turacin, an animal pigment containing copper, and several mineral species, including the only British cerium mineral. He published many books on agricultural chemistry, including the well-known "Laboratory Guide for Agricultural Students," which has run into eight editions. He was elected a Fellow of the Royal Society in 1888, and was created K.C.V.O. in 1909.

## Journal and Patent Literature.

PATENT SPECIFICATIONS may be obtained by post by remitting as follows:—

English.—6d. each, to the Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

United States.—1s. each, to the Secretary of the Society.

French.—1 fr. 05 c. each as follows: Patents dated 1902 to 1907 inclusive, Delin et Cie., Rue Ferou 8, Paris, (56); Patents from 1908 to date, L'Imprimerie Nationale, 87, Rue Vieille du Temple, Paris.

### I.—GENERAL PLANT; MACHINERY.

*Pyrometry; Recent progress in* — C. R. Darling, J. Roy. Soc. Arts, 1915, 63, 590—607.

SINCE 1910 no pyrometers based upon new principles have been introduced, and the advances made have been confined to improvements in methods previously in use. The values now generally recognised for purposes of standardisation are:—Water (b. pt.), 100° C.; aniline (b. pt.), 184°; naphthalene (b. pt.), 218°; tin (m. pt.), 232°; cadmium (m. pt.), 321°; lead (m. pt.), 327°; zinc (m. pt.), 419°; sulphur (b. pt.), 445°; antimony (m. pt.), 631°; sodium chloride (m. pt.), 800°; silver (m. pt. in reducing atmosphere), 961°; gold (m. pt.), 1063°; copper (m. pt., graphite covered), 1083°; lithium metasilicate (m. pt.), 1202°; nickel (m. pt.), 1450°; palladium (m. pt.), 1550°; platinum (m. pt.), 1755°; tungsten (m. pt.) about 3000°; carbon arc, about 3500° C.

*Thermo-electric pyrometers.*—Base metals have been used to a very considerable extent for the construction of thermo-couples. This has been due not only to the comparative cheapness of such materials, but to the fact that properly-chosen base-metal couples develop a relatively high E.M.F. Kowalke (this J., 1912, 1010; 1915, 379) has shown that base-metal couples are liable to alter if subjected to continued heating, and suggests that such couples should be "aged" by continued heating before calibration. Carbon and graphite couples have been used for temperatures exceeding 1200° C. (Bidwell, Phys. Rev., June, 1914). The potentiometer principle has been largely adopted for commercial instruments.

*Resistance pyrometers.*—Few changes have been introduced. Sir Wm. Crookes has shown recently (this J., 1912, 540) that platinum is measurably

volatile above 1000° C., and this explains why pyrometers give erroneous readings when continuously used for higher temperatures.

*Radiation pyrometers.*—There has been a considerable increase in the use of these instruments, and Whipple has applied the Féry pyrometer to the determination of the temperature of molten metals by mounting it at the open end of a fire-clay tube, which is dipped into the molten metal, thus keeping the couple in permanent focus.

*Optical pyrometers* have been improved in detail and a new type depending upon the principle of colour extinction has been introduced. Lovibond has introduced a colour-matching pyrometer which has been found to be very sensitive.

*Recorders.*—Instruments which give an inked record are taking the place of the photographic recorder of Roberts-Austen.—W. H. C.

*Silica dish: Heat transmission capacity of a* — W. K. Lewis, J. Ind. Eng. Chem., 1915, 7, 110—111.

SOLUTIONS of boiling point ranging from 100° C. (water) up to 240° C. (concentrated zinc chloride) were evaporated in a silica dish set in an ordinary gas crucible furnace, and the heat consumption was calculated from the quantity of water required to keep the volume of the solution constant during evaporation. The results obtained are plotted in curves, and it is shown that the heat absorbed and transmitted by the dish per unit area and time is equal to the sum of two terms, the first of which expresses the quantity of heat transmitted by conduction from the furnace gas and is proportional to the difference in temperature (degrees Centigrade) between the gas and the dish, whilst the second represents the heat transmitted by radiation from the furnace walls, and is proportional to the difference between the fourth

powers of the absolute temperatures of the walls and of the dish. Similar relations hold for any unglazed ceramic material, and therefore in designing fuel-fired furnaces, the aim should be to provide the greatest possible area of furnace wall, so placed as to obtain effective contact with the fire gases and the maximum direct radiation to the heat-absorbing surface. The latter object will be best attained by filling the flue with open checkerwork refractory material of high heat-conductivity, so that heat absorbed from the gases will be transmitted rapidly to a surface radiating directly to the object to be heated. In the concentration of sulphuric acid in silica dishes arranged in series, it is recommended that a thick fuel bed be used with insufficient air for complete combustion, and also with injection of steam, if necessary, further to reduce the temperature to which the dishes set directly over the fire are exposed. The carbon monoxide or water-gas produced is burnt by introducing air at different points along the flue, which is provided with heat-conducting refractory material to facilitate transmission of heat to the dishes by radiation. In this way breakage of dishes is minimised and it is possible to maintain at all parts of the flue the highest temperature the dishes can withstand without danger.—A. S.

*Fractional distillation with regulated stillheads.* M. A. Rosanoff, J. F. W. Schulze, and R. A. Dumphy. J. Amer. Chem. Soc., 1915, 37, 1072—1079. (See this J., 1915, 301.)

As the regulated stillhead may possibly find industrial application, particularly for the fractionation of liquid mixtures containing three or more constituents, experiments were carried out on a ternary mixture of toluene, carbon tetrachloride, and ethylene bromide. In distillations with a stillhead kept at a constant temperature, the composition of the distillate at any moment is the same as that of the vapour evolved by a mixture whose boiling point is equal to the stillhead temperature. The composition of the distillate from three or more substances depends upon that of the original mixture, and (unlike that of binary mixtures) varies slightly during the course of a single distillation; but the variation shown by successive distillates becomes less and less as the stillhead temperature approaches the boiling point of the most volatile constituent. Practically, therefore, the regulated stillhead can be used to obtain a uniform distillate from a ternary or more complex mixture.—E. H. T.

#### PATENTS.

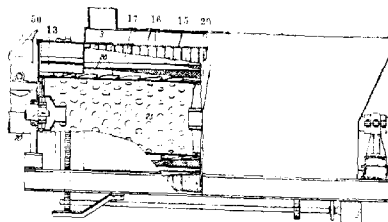
*Distilling, evaporating, or concentrating liquids or solutions: [Electrically heated] apparatus for* — J. Leopold-Brodie, London. Eng. Pat. 1123, Jan. 15, 1914.

The calandria or heating belt of the still or evaporating pan is so constructed that it may be removed bodily from the apparatus. The tubes of the heating belt each contain or are surrounded by a resistance coil through which an electric current is passed. The individual heating tubes, together with the resistances, can also be easily removed. —W. H. C.

*Earthy materials [for paving, etc.]: Heating, drying, and pulverising* — M. A. Popkess, New York. Eng. Pat. 9281, April 14, 1914.

The material is fed through a hopper, 50, a measuring device, and a screw conveyor, to the lifting shelves, 13, of the slowly rotating drum, 3. It then drops on the spiral blades, 26, attached to the perforated drum, 25, and is pulverised between the blades, 26, and shelves, 17, while moving forward. The fine material passes through

the tapering screen, 15, to the spiral conveyor blades, 16, which discharge it. A liquid fuel burner, 70, injects hot gases through the drum,



25, and surrounding chamber, the gases finally returning through the annular space, 20, between the fixed casing and rotating drum.—W. F. F.

*Solvents: Apparatus for recovering* — E. C. R. Marks, London. From E. I. du Pont de Nemours Powder Co., Wilmington, Del., U.S.A. Eng. Pat. 10,351, April 27, 1914.

The material containing the solvents to be recovered is placed in or passed through a shallow evaporation chamber, near the bottom, while heating coils are fixed near the top but close to the material. The chamber forms part of a closed air circuit; the hot air and vapours pass from it down a vertical passage to a condenser, from which the condensed solvent is drawn off, and the air then passes upwards over heating coils back to the evaporation chamber. The air leaving the evaporating chamber may be diverted upwards (through a baffle chamber, to increase the draught, before passing down to the condenser.—W. F. F.

*Centrifugal apparatus for the treatment or extraction of liquids.* T. K. Irwin, Westminster, Eng. Pat. 10,810, May 1, 1914.

The material is thrown from a series of horizontal rotating discs mounted on a vertical shaft, on to the porous walls of a concentric cylinder also carried by the shaft, from which it is removed by cutters adjustable vertically and laterally. The material falls through the inclined radial cutting arms at the base of the cylinder into a receiver provided with a conveyor. The apparatus is more suitable for dealing with materials of small liquid content than that described in Eng. Pats. 1925 and 3526 of 1913 (this J., 1914, 274).—W. F. F.

*Cooling towers.* F. C. Schmidt, London. Eng. Pat. 21,205, Oct. 19, 1914.

The hot liquid trickles down over cooling hurdles divided by vertical partitions which have horizontal extensions, each forming a collecting tray for its own section only. The trays are arranged in steps so that air can be admitted from beneath the tower.—W. F. F.

*Mixtures of liquids and solids: Apparatus for treating [filtering]* — A. E. Vandercook, Alameda, Cal., Assignor to California Macvau Co., San Francisco, Cal. U.S. Pat. 1,135,080, April 3, 1915. Date of appl. March 3, 1914.

The pulp being filtered is projected directly on to the surface of the filtering medium from apertures in a cross of pipes, which is rotated below the level of liquid pulp in the filter tank, and just above the surface of the filter medium.—W. H. C.

*Drying apparatus.* A. Tegoli, Assignor to C. R. Bertoli and L. Tantimonaco, Santa Rosa, Cal. U.S. Pat. 1,135,536, April 13, 1915. Date of appl. Sept. 15, 1914.

The dryer has a middle and two lateral chambers.

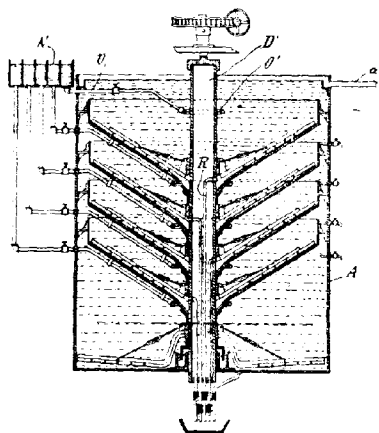
Air currents produced by blast fans in the lateral chambers and an exhaust fan in the middle one are regulated by perforated drawers arranged in drawer spaces above each chamber.—W. H. C.

*Separator.* W. K. Liggett, Columbus, Ohio, Assignor to The Jeffrey Manufacturing Co. U.S. Pat. 1,135,594, April 13, 1915. Date of appl., Feb. 18, 1907.

PULVERISED material is withdrawn from a disintegrator by a current of air and delivered into the first of two settling chambers, where the coarser particles settle. The finer particles pass along with the air into a second and larger conical settling chamber; the finely divided product settles and is withdrawn through a valved opening in the bottom of the chamber, and the air passes through a screen in this chamber and is returned to the disintegrator. The coarser particles from the first settling chamber are delivered into the return air pipe, and are conveyed back to the disintegrator.—W. H. C.

*Separation of finely-divided solids from liquids; Apparatus for the —.* J. V. N. Dorr, Denver, Colo. U.S. Pat. 1,135,997, April 20, 1915. Date of appl., June 5, 1913.

THE pulp is fed from the trough, N, through the pipes, O, O', to the space above the superposed conical trays contained in the tank, A. "Sweeps"



attached to the central hollow shaft, D', are rotated above the surface of the trays and sweep the settled solids to the centre and discharge them through the pipes, R. The clarified liquid is discharged through the overflow, a.—W. H. C.

*Classifying comminuted material; Process of and apparatus for —.* H. M. Sutton, W. L. and E. G. Steele, Dallas, Tex. U.S. Pat. 1,136,293, April 20, 1915. Date of appl., Aug. 13, 1913.

THE particles are projected upwards intermittently along an inclined, reticulated surface. In the intervals the particles tend to pass downwards owing to the action of gravity. The separated particles are collected separately.—W. H. C.

*Solids from liquids; Process of obtaining the —.* O. E. Merrell, Assignor to Merrell-Soule Co., Syracuse, N.Y. U.S. Pat. 1,136,356, April 20, 1915. Date of appl., Feb. 16, 1912.

THE liquid is introduced at the vortex of a forward-moving spiral current of air which sprays and vaporises it. The solid constituents are subsequently separated from the moisture-laden air in the form of a dry powder.—W. F. F.

*Chemical reactions at high temperatures; Process for effecting —.* F. Meyer, Ger. Pat. 281,001, Feb. 5, 1914. Addition to Ger. Pat. 261,922.

INSTEAD of blowing the material under treatment through a "reversed flame" as described in the chief patent (this J., 1913, 900), the flame, preferably under increased pressure, is directed on to the material. The process is applicable to the preparation of nitrides from oxides, the reduction of oxides to metals, and the melting of metals.—A. S.

*Filtering apparatus.* W. A. Stedman, Womb. Nev., U.S.A. Eng. Pat. 9919, April 22, 1911. Under Int. Conv., Aug. 13, 1913.

SEE U.S. Pat. 1,100,267 of 1911; this J., 1911, 781.

*Drying machine.* J. McL. Cameron, London, U.S. Pat. 1,136,933, April 27, 1915. Date of appl., April 25, 1914.

SEE Eng. Pat. 10,467 of 1913; this J., 1915, 260.

*Dryer.* H. P. Coe, Painesville, Ohio, Assignor to A. S. Williams, Long Island City, N.Y. U.S. Pat. 1,138,086, May 4, 1915. Date of appl., Aug. 7, 1912.

SEE Eng. Pat. 16,712 of 1913; this J., 1914, 72.

*Mixing machine.* A. A. Warner, Assignor to Landers, Frary, and Clark, New Britain, Conn. U.S. Pat. 1,137,637, April 27, 1915. Date of appl., March 25, 1914.

SEE Eng. Pat. 16,753 of 1914; this J., 1915, 261.

*Mixing machine.* R. Tiedtke, Assignor to Farb- und Form-Meister, Lucius, u. Brüning, Hoechst, Germany. U.S. Pat. 1,139,085, May 11, 1915. Date of appl., Oct. 27, 1910.

SEE Eng. Pat. 4819 of 1908; this J., 1908, 674.

*Gaseous mixture; Process and device for separation of the elements composing a — by centrifugal action.* E. Mazza, Turin, Italy. U.S. Pat. 1,137,774, May 4, 1915. Date of appl., May 25, 1911.

SEE Fr. Pat. 430,621 of 1911; this J., 1911, 1306.

*Refrigerating apparatus.* H. H. Southworth, Cleveland, Ohio, and F. W. Wolf, Chicago, Ill. Assignors to Iceless Machine Co., Cleveland, Ohio. U.S. Pat. 1,139,160, May 11, 1915. Date of appl., Oct. 25, 1912.

SEE Eng. Pat. 27,598 of 1912; this J., 1914, 405.

*Stills or evaporating plant for the distillation of tar, pitch, oil, and like heavy liquids or viscous substances.* Eng. Pat. 19,392. See III.

*Pyrometer.* Eng. Pat. 7597. See XXIII.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coke; Effect of different methods of crushing on the determination of the ash of —.* F. A. Eastaugh, Inst. Min. and Met., May 20, 1915. (Advance proof.) 3 pages.

FOUR samples of coke were prepared by different methods. No. 1 was broken by means of a wooden mallet, reduced in size by quartering, then ground in a stone mortar to pass a 60-mesh screen. No. 2 was broken on an iron plate by means of a bucking hammer and reduced to 60-mesh on a bucking plate. No. 3 was broken in the same manner as No. 2, but the final grinding was done in a heavy Weatherhead mill. No. 4 was passed through Taylor crusher, and ground in a Braun disc mill.

Two grams were taken from each dried sample and burnt, and the amount of iron oxide determined in the ash with the following results:—

No.	Weight of ash.	Weight of $\text{Fe}_2\text{O}_3$ in ash.	Percentage of ash.	
			Uncorrected.	Corrected for $\text{Fe}_2\text{O}_3$ .
	gm.	gm.		
1	0.6558	0.4093	17.79	17.79
2	0.4156	0.0746	20.78	17.52
3	0.3916	0.0432	19.58	17.88
4	0.3650	0.0321	18.25	17.11

It was assumed that the amount of iron found in the ash of No. 1 was a natural constituent of the coke, and the other results were corrected on this basis. No. 4 seems to be the best result, the amount of foreign matter introduced during the crushing being probably less than in the other three samples.—H. R. D.

*Gas; Removal of carbon bisulphide from* —. Teun. Het Gas, April, 1915. J. Gas Lighting, 1915, 130, 331.

EXPERIMENTS were made to test the accuracy of an earlier statement that 40 to 70% of the carbon bisulphide could be removed by passing the gas through spent oxide having a high sulphur content. With spent oxide containing 32.3% S. and with varying velocity and temperature of gas, up to about 12% of its sulphur was removed from the gas. The same result was obtained with pure powdered sulphur, thus showing that no practical purification can be based on this method.—W. F. F.

*Methane-air mixtures; Influence of temperature and pressure on the explosibility of* —. G. A. Burrell and I. W. Robertson. J. Ind. Eng. Chem., 1915, 7, 117-419.

The mixtures were ignited in a 100 c.c. glass explosion pipette by means of a spark produced between two platinum wires sealed into the upper part. The pipette was heated to the desired temperature by an electrically heated oven. The lower limit was not changed appreciably by increase of pressure up to 5 atmospheres, but was reduced by rise of temperature from 5.5%  $\text{CH}_4$  at the ordinary temperature to 4.98-5.15% at 200 C. and 3.75-4%  $\text{CH}_4$  at 500 C.—A. S.

*Gasoline vapour and air; Inflammable limits of mixtures of* —. G. A. Burrell and H. T. Boyd. J. Ind. Eng. Chem., 1915, 7, 111-117.

The lower limit of inflammation of mixtures of vapour from gasoline of 73 B. (sp. gr. 0.696) and air was found to be 1.9-2.4% in a 100 c.c. Hempel explosion pipette when the mixture was ignited from the top, and 1.5-1.6% when ignited from below by means of a spark from an induction coil; when ignited from the bottom in a bottle of 2800 c.c. capacity by means of a flash produced by drawing apart two wires through which a current of 7 amp. at 220 volts was flowing, the lower limit was 1.1-1.5%. Under the last-mentioned conditions the upper limit was 6.0-6.1%, as compared with 5.2-5.3% in the Hempel pipette with ignition at the top. Similar results were obtained with vapour from "cleaners' naphtha" of 59-60 B. (sp. gr. 0.715-0.749). When the temperature of the mixture was raised before igniting, the lower limit gradually decreased until with an initial temperature of 400 C. it had the value 1.02-1.22%.—A. S.

*Petrol substitutes in internal combustion engines. Velocity of vaporisation.* D. Meneghini. Annali Chim. Appl., 1915, 3, 235-244.

A CURRENT of dry air was drawn through the liquid

under examination contained in a U-tube kept in a thermostat at 25 C. and the amount of liquid vaporised was ascertained from the loss in weight. The duration of each test was 5 mins. and the velocity of the air current was such that 3 litres passed in this period. Under these conditions the percentage loss of weight by the different liquids examined was: Petrol of sp. gr. 0.700, 20.5; 99% alcohol, 1.99; 95% alcohol, 1.90; 90% and 80% alcohol, 1.85; methylalcohol, 3.60; benzene, 5.10; toluene, 1.73. Three samples of 90% benzol gave the following results:—I. (81.6% below 85 C., 99.6% below 105 C.), 5.35; II. (77.7% below 85 C., 96.9% below 105 C.), 5.25; III. (51.1% below 85 C., 92.1% below 105 C.) 5.0. Mixtures of alcohol and benzol gave higher values than either of the components singly. For mixtures containing 40% of alcohol by volume the results with the different specimens of 90% benzol were: I., 6.25; II., 6.30; III., 5.39; for 20% alcohol mixtures: I., 6.30; II., 6.55; III., 5.55; for 35% alcohol mixtures: I., 6.40; II., 6.55; III., 5.99; and for 50% alcohol mixtures: I., 6.15; II., 6.29; III., 5.80.—A. S.

#### PATENTS.

*Peat; Utilisation of* —. M. A. Adam, and Wet-carbonizing Ltd., London. Eng. Pat. 3888, Feb. 11, 1914.

WET-CARBONISED peat which has been de-watered by pressure to a semi-solid mass, is subjected to a pressure of about 250 lb. per sq. in. in a filter-press with collapsible chambers, while a current of about 50 amps. per sq. ft. at about 50 volts is passing through it. (See also Eng. Pats. 17,610 of 1911 and 25,146 of 1912; this J., 1912, 1171 and 1914, 316.)—W. F. F.

*Peat briquettes; Production of wet-carbonised* —. N. Testrup, London, and T. Rigby, Dumfries. Assignors to Wetcarbonizing Ltd., London. U.S. Pat. 1,439,315, May 11, 1915. Date of appl., May 6, 1912.

SEE Eng. Pat. 11,551 of 1911; this J., 1912, 675. The wet-carbonised peat, after being filter-pressed, is disintegrated, dried to a water-content of about 5%, and heated to not above 100 C. before being consolidated by pressure applied suddenly.

*Coke, coal, or like granulated substances; Mechanism for screening or sifting* —. Drakes Ltd., Halifax, and A. Walker, Wallsend-on-Tyne. Eng. Pat. 8439, April 3, 1914.

The material is fed down an inclined reciprocating trough, the bottom of which has a series of combs with pointed fingers pointing down the trough, and having a slight adjustable upward inclination. A rotary cylindrical apparatus may be similarly fitted.—W. F. F.

*[Gas] retorts; Apparatus applicable for use in discharging* —. Drakes Ltd., Halifax, and A. Walker, Wallsend-on-Tyne. Eng. Pat. 8438, April 3, 1914.

A PORTABLE framework placed at the mouths of a battery of horizontal retorts carries inclined guides which form a single zig-zag path leading from the successive tiers of retorts to a common discharge point.—W. F. F.

*Petroleum; Treatment of* —. J. A. Dubbs, Santa Monica, Cal., Assignor to National Hydrocarbon Co., Chicago, Ill. U.S. Pat. 1,135,506, April 13, 1915. Date of appl., June 3, 1911.

PETROLEUM is mixed with water under pressure, and the mixture is vaporised, also under pressure. The pressure is then relieved, and the vaporised mixture discharged into a heated chamber in the

form of fine spray. The vapours are subsequently condensed.—W. F. F.

*Liquid hydrocarbons; Treatment of— for their purification and the production of motor spirit and by-products.* J. J. Shedlock, and Optime Motor Spirit Synd., Ltd., London. Eng. Pat. 1878, Jan. 23, 1914.

THE oil is first freed from pitch, etc., by passing through a vessel containing caustic alkalis or metallic oxides heated to 300°–400° F. (150°–205° C.), and then agitated with an emulsion of oil and 10–20% water. The mixture passes through a vessel containing a catalyst such as iron, steel, or nickel, heated to 500°–900° F. (260°–480° C.) according to the oil to be treated, and the resulting oil and gas then pass to an expansion chamber where the light vapour is removed and liquefied to form a motor spirit. The permanent gases may be used as fuel, and the residual heavy oil may be further treated as desired.—W. F. F.

*Distilling coal; Method of—* W. J. Mellersh-Jackson, London. From International Gas Development Co., New York. Eng. Pat. 9849, April 21, 1914.

SEE U.S. Pat. 1,097,513 of 1914; this J., 1914, 685.

*Petroleum; Apparatus for the industrial manufacture of a new spirit by the isomerisation of—* A. Testelin, Laeken, and G. Renard, Ixelles, Belgium. U.S. Pat. 1,138,260, May 4, 1915. Date of appl., Aug. 20, 1908.

SEE Fr. Pat. 393,551 of 1908; this J., 1909, 133.

*Lubricants.* H. Wade, London. From The Pensacola Tar and Turpentine Co., Gull Point, Fla., U.S.A. Eng. Pat. 18,235, Aug. 1, 1914.

SEE U.S. Pat. 1,109,298 of 1914; this J., 1914, 1002.

*Process for obtaining ammonia from coke breeze.* Ger. Pat. 281,996. See VII.

*Detecting, indicating, and recording the presence and proportion of [inflammable] gas in the atmosphere of mines.* Eng. Pat. 9196. See XXIII.

*Calorimetric apparatus. Calorimeter. Combustion device.* U.S. Pat. 1,136,359–1,136,361. See XXIII.

[Gas] calorimeter. U.S. Pat. 1,136,884. See XXIII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Peat; Utilisation of—* T. Rigby, Dumfries, and Wetcarbonizing, Ltd., London. Eng. Pat. 1676, Jan. 21, 1914.

PEAT which has been subjected to heat treatment such as that described in Eng. Pat. 17,610 of 1911 and 25,146 of 1912 (this J., 1912, 1471, and 1914, 346) is distilled, and by-products such as wax are recovered from the distillation gases. The residue is gasified in a producer, or part may be used as solid fuel, and the gas is treated separately for the recovery of by-products, its heat being used for the distillation process. The producer and still gases, after recovery of by-products, may be mixed and used as fuel.—W. F. F.

*Decoloriser [from argol]; Process for making a—* F. W. Spanutius, Assignor to Pan Chemical Co., Hastings-upon-Hudson, N.Y. U.S. Pat. 1,135,216, April 13, 1915. Date of appl., Feb. 7, 1912.

ARGOL is treated with caustic soda to decompose the colouring matter, potassium chloride is added, and the residue, consisting of 50% moisture, and 50% crude fibre of argol, calcium salts, and sand is separated from the solution. The residue is heated in a retort until carbonisation is complete, the evolved vapour being allowed to escape.

—B. N.

*Neon vacuum tubes; Manufacture of—* G. Claude, Boulogne-sur-Seine, France. Eng. Pat. 2020, Feb. 8, 1915. Under Int. Conv., Feb. 12, 1914.

THE tube to be formed is connected with one or more receptacles containing a purifying agent such as charcoal, and these are cooled by means of liquid air, hydrogen, oxygen, or nitrogen. When the neon in the vacuum tube has become contaminated with impurities liberated by the passage of the current, the gas is circulated over the purifying agent so as to absorb the neon and impurities, the neon being afterwards liberated in a pure condition. The gas may be transferred to and from the purifying agent (1) by cooling a single receptacle alternately with liquid hydrogen and liquid air; (2) by cooling two receptacles at opposite ends of the vacuum tube with liquid oxygen and liquid nitrogen and then transposing the cooling agents so as to produce circulation of the neon from one receptacle to the other; (3) by expanding and contracting the gas by the heating effect of the current, or by alternately diminishing and increasing the pressure in the vacuum tube by means of a pressure vessel containing mercury, or by two pressure vessels connected one to each end of the vacuum tube. In this latter case the gas may be circulated over heated lithium and the like instead of cooled charcoal. The neon may also be supplied to the vacuum tube through a cooled receptacle containing charcoal to facilitate the formation. (See also this J., 1911, 13, 736.)—B. N.

*Wood; Destructive distillation of—* A. Cameron, East Jordan, Mich. U.S. Pat. 1,137,255, April 27, 1915. Date of appl., Sept. 26, 1913.

THE wood is packed into a sealed retort having horizontal supporting members at different levels, so that as the charge shrinks during distillation, separate horizontal layers of charcoal are formed with intervening circulation spaces.—W. F. F.

*Wood charcoal; Apparatus for making—* L. Fédizet, Assignor to Huilerie et Savonnerie de Lurian, Bouches-du-Rhône, France. U.S. Pat. 1,137,852, May 4, 1915. Date of appl., July 21, 1913.

SEE Fr. Pat. 409,916 of 1909; this J., 1910, 800.

## III.—TAR AND TAR PRODUCTS.

*Vacuum tar; Saturated hydrocarbons of—* A. Pictet and M. Bouvier. Comptes rend., 1915, 160, 629–631. (See also this J., 1913, 1098; 1914, 70; 1915, 163.)

THE unsaturated hydrocarbons from "vacuum tar" were separated from the saturated members by absorption in liquid sulphur dioxide. Fractional distillation of the unabsorbed (saturated) hydrocarbons at atmospheric pressure resulted in the isolation of four additional hydrocarbons of the general formula,  $C_{11}H_{22}$ , all of which were unaffected by bromine or permanganate. The following constants of the hydrocarbons are practically identical with those quoted by Mabery (this J.,

1897, 727; J. Amer. Chem. Soc., 1903, 25, 267; 1911, 33, 264) for hydrocarbons obtained from Canadian and Californian petroleum:—

Formula.	B. pt. (730 mm.) ° C.	Sp. gr.	Refractive index at the same temperature.
$C_6H_{12}$	135—137	0.7530 at 20° C.	1.4212
$C_{10}H_{20}$	160—161	0.7680 at 20°	1.4272
$C_{12}H_{24}$	211—213	0.7862 at 21°	1.4293
$C_{14}H_{28}$	227—229	0.7953 at 20°	1.4379

The substance,  $C_6H_{12}$ , is hexahydromesitylene; and all the members of the series are regarded as homologues of cyclohexane. Upon continuing the distillation of the vacuum tar above 240° C. under reduced pressure (15 mm.), the distillate solidified partially upon cooling, and after washing with cold acetone and repeated crystallisation from the same solvent, colourless needles were obtained melting at 62—63° C. and having the sp. gr. 0.9128 at 25° C. This saturated hydrocarbon,  $C_{20}H_{40}$ , belongs to the above series and is not formed during distillation, but pre-exists in the coal; it also occurs in Galician and other natural petroleum and is identical with the so-called melene, obtained by Brodie in 1849 by the dry distillation of beeswax.—J. R.

**Aldehydes:** Action of — on the Grignard reagent. J. Marshall. Chem. Soc. Trans., 1915, 107, 509—523.

WHEN excess of an aliphatic or aromatic aldehyde is added to the Grignard reagent, this excess acts as an oxidising agent and converts the carbonyl derivative, which is first formed, into a ketone, being itself reduced to a primary alcohol. Thus benzaldehyde (2 mols.) reacts with magnesium-phenyl bromide (1 mol.) giving benzophenone and benzyl alcohol. If excess of magnesium is present, either due to the formation of diphenyl or purposely added, reduction of diphenylcarbinol to tetraphenylethane or diphenylmethane takes place. The mechanism of the reaction is probably similar to that of the formation of benzoic acid and benzyl alcohol from benzaldehyde in presence of sodium hydroxide. It is suggested that 2 mols. of water are eliminated successively from 2 mols. of the hydrate of benzaldehyde, with formation of a derivative of ethylene oxide, which adds on a molecule of sodium hydroxide to give an ortho-compound yielding benzyl alcohol and benzoic acid on hydrolysis. When ethyl acetate acts on magnesium carbonyl haloids, a good yield of the carbonyl acetate is obtained. T. C.

#### PATENTS.

*Stills or evaporating plant for the distillation of tar, pitch, oil, and like heavy liquids or viscous substances.* Sir F. K. I. Crossley and H. W. Brighton. Manchester. Eng. Pat. 19,392, Sept. 3, 1914.

THE neck of the still is lagged with asbestos, and has a constricted opening controlled by a valve whose position can be adjusted and indicated. A low-pressure safety valve is also provided. W.F.F.

*Preparation of a pure, pale coumarone-resin from heavy benzols of b. pt. 160—180° C.* Ger. Pat. 281,432. See XLIII.

#### IV.—COLOURING MATTERS AND DYES.

*Orcinolphthaleins, orcinoltetrachlorophthaleins, and their derivatives.* W. R. Orndorff and E. R. Allen. J. Amer. Chem. Soc., 1915, 37, 1201—1258.

THE methods of preparing and separating the isomeric orcinolphthaleins have been improved

and the influence of conditions of preparation on the amounts of isomers formed has been studied. *o*-Orcinolphthalein only exists in the colourless lactoid form. An explanation of the colour changes of *o*-orcinolphthalein with alkalis is advanced which is more in accord with the facts than that of Baeyer (Annalen, 1910, 372, 120). With caustic potash the first violet salt formed is the *o*-quinonoid monopotassium salt, the colourless form of which is the monopotassium salt of *o*-orcinolphthaleincarbinolcarboxylic acid; the blue salt is the di- and not the tetra-potassium salt. Both the colourless unstable lactoid and coloured stable *p*-quinonoid forms of the  $\beta$ -compound have been prepared. The  $\gamma$ -phthalein is only known in the coloured form, which behaves as a dimethylfluorescein, and hence has a *p*-quinonoid structure, and not the *o*-quinonoid structure assigned to it by Kehlmann (Ber., 1912, 45, 3505).  $\gamma$ -Orcinolphthalein has both acid and basic properties, combining with dry ammonia gas to form a coloured diammonium salt and with dry hydrochloric acid gas to form a coloured hydrochloride. All the isomers form coloured mono-hydrochlorides. Colourless hydrates, acetyl and benzoyl derivatives, and compounds with solvents such as methyl alcohol have been prepared. The three tetrabromo-orcinolphthaleins and the three orcinoltetrachlorophthaleins are colourless lactoid compounds, the bromine atoms in the former case being in the two orcinol residues, exactly as in the case of Eosin. The tendency to assume the free quinonoid form is reduced by the introduction of halogen into the phthalein molecule. This is in agreement with the observations of Orndorff and Hitch (J. Amer. Chem. Soc., 1911, 33, 680) that fluorescein in the free state exists only in the coloured quinonoid form, whereas tetrachlorofluorescein exists in a more stable coloured and a colourless form; Eosin is probably a mixture of the two forms, while free tetrachloro-eosin is colourless, and the coloured modification has not yet been prepared.—F. W. A.

*Coal tar products and dyes in America.* T. H. Norton. U.S. Bureau of Commerce, April, 1915.

DURING the past few months there has been great advance in the production of dyestuffs. Efforts are being made to increase the output of benzol, and contracts have been placed by one company for 92 additional by-product coke-ovens, with complete equipment for benzol recovery, at an outlay of about £200,000. It is expected that the daily production of benzol in the Pittsburgh district will soon exceed 20,000 gallons. The recovery works of the United States Steel Corporation at Erie (Ind.) are producing 12,000 gallons of benzol per day. Additional recovery plants are under construction in connection with the coke-works of the Republic Iron and Steel Company at Youngstown (Ohio), of the Lackawanna Steel Company at Buffalo (N.Y.), and of the Inland Steel Company at Inland Harbor (Ind.). The Benzol Products Company have nearly completed at Marcus Hook (Pa.) extensive plant which is to be devoted to the manufacture of intermediates on a large scale. It is hoped to produce in these works most of the aniline oil and salts required by American colour-works. Another company has been established for the manufacture of sulphur colours. The Cambria Steel Company is planning to erect at once plant to manufacture dyestuffs, using as raw material the 6 tons of benzol and its homologues yielded daily by their coke plant. Several firms commanding ample capital are maturing plans for establishing new plants for the production of artificial dyestuffs. The existing American factories for manufacturing coal-tar dyes are making every effort to increase their output, though much hampered by difficulty in

obtaining raw material during the last few months. It is regarded as certain that production will grow rapidly as more ample supplies of benzol, etc., are assured, and various obstacles incident to the complete cessation of the import of intermediate products from Europe are overcome.

*Dyeing values of natural Indian dyestuffs.* Srivastava. See VI.

*Mercury derivatives of aromatic amines. I. Structure of primary and secondary p-aminophenylmercuric compounds.* Jacobs and Heidelberg. See XX.

#### PATENTS.

*Selenium dyestuff corresponding to Methylene Blue: Isolation of the —.* W. Fraenkel. Ger. Pat. 280,713, Aug. 16, 1913.

THE product obtained by treating *p*-phenylenediamine or the like with hydrogen selenide or other substance capable of yielding selenium, and then oxidising the mixture, is shaken with phenol, and the phenol solution is separated and shaken with aqueous hydrochloric acid and a solvent for the phenol immiscible with water, *e.g.*, ether or carbon tetrachloride. The dyestuff is converted into its hydrochloride, which dissolves in the aqueous solution and can be recovered by distilling off the water and hydrochloric acid under diminished pressure. The only practical application of the dyestuff is to therapeutic purposes, for which its isolation in the form of its zinc chloride double salt is inadmissible.—A. S.

*Arglaminanthraquinone dyestuffs and process of making same.* G. Kränzlein, R. Hagenbach, and F. Giloy, Assignors to Farb- u. vorm. Meister, Lucius, u. Brünig, Höchst, Germany, U.S. Pat. 1,138,670, May 11, 1915. Date of appl. May 13, 1914.

SEE Fr. Pat. 472,100 of 1914; this J., 1915, 417.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### PATENTS.

*Cellulose compounds (alkali-cellulose): Manufacture of —.* Courtaulds, Ltd., London, W. H. Glover, Braintree, and L. P. Wilson, Coventry. Eng. Pat. 13,055, May 27, 1914.

ALKALI-CELLULOSE is matured by treating the mixture of cellulose and alkali with an oxidising agent, such as a soluble peroxide, a hypochlorite, or a current of oxygen alone or mixed with inert gases at above 30° C. The product is particularly adapted for the manufacture of artificial silk by the viscose process.—F. Sp.

*Cellulose compounds: Manufacture of —.* Courtaulds, Ltd., London, and L. P. Wilson, Coventry. Eng. Pat. 14,975, June 18, 1914.

A CATALYTIC agent such as an oxide or hydroxide of iron, nickel, cobalt, cerium, or vanadium, or a mixture of these, is employed to assist the maturing of alkali-cellulose by oxidation, previous to its conversion into cellulose xanthate (viscose).—B. N.

*Sugar cane: Process of treating —.* [Utilisation of bagasse for paper-making.] T. Lee, Dedham, Mass., U.S.A. Eng. Pat. 17,831, July 28, 1914. Under Int. Conv., Sept. 16, 1913.

THE bagasse as it comes from the mills is partially dried and then beaten or otherwise disintegrated,

*e.g.*, in a Williams pulveriser, so that the pith and cortex can be afterwards separated from the rest of the tissue ("selected fibre") which is well suited for paper-making. The granular pith, for example, may be removed by means of a screen of  $\frac{1}{4}$  in. mesh, and the selected fibre may be separated from the strips of cortex by a coarser screen. The rejected tissues, which usually exceed 50% of the woody matter of the bagasse, are used as fuel, and the selected fibre is washed and digested, *e.g.*, for 8 hours under a steam pressure of 40 lb. with caustic lye equivalent to 14% NaOH of the weight of the fibre.—J. H. L.

*Pyroxylin bodies: Process of forming —.* F. Kniffen, Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, Del., U.S. Pat. 1,135,026, April 13, 1915. Date of appl. April 12, 1913.

PYROXYLIN products are formed by evaporating a solution of pyroxylin in a mixture of ethyl acetate and benzene.—F. Sp.

*Acetylcellulose plastic compounds: Process of making —.* W. G. Lindsay, Caldwell, N.J., Assignor to The Celluloid Co., New York, U.S. Pat. 1,136,248, April 20, 1915. Date of appl. Nov. 27, 1911.

PLASTIC masses are produced by incorporating acetone-soluble acetylcellulose with tetrachloro-ethylacetanilide and a small proportion of methyl alcohol, *e.g.*, about one to one and a half times the quantity of tetrachloro-ethylacetanilide.—F. Sp.

*Films from plastic material: Manufacture of —.* J. E. Brandenberger, Thion-les-Vosges, France. Eng. Pat. 13,072, May 27, 1914. Under Int. Conv., May 31, 1913.

SEE Fr. Pat. 458,638 of 1913; this J., 1913, 1063.

*Esters of cellulose: Manufacture of —.* Soc. Chim. des Usines du Rhône, Paris. Eng. Pat. 13,696, June 5, 1914. Under Int. Conv., June 25, 1913.

SEE Fr. Pat. 473,399 of 1914; this J., 1915, 488.

*Extraction of mineral salts and organic constituents of pulp.* U.S. Pat. 1,136,390. See VII.

*Process for graining and waterproofing artificial leather [and paper, textiles, etc.].* Ger. Pat. 281,302. See XV.

*Manufacture of useful products [celluloid substitutes, etc.] from vinyl esters.* Ger. Pat. 281,687 and 281,688. See XIII.

*Production of a plastic material suitable for the preparation of a horn substitute, films, threads, lacquers, etc.* Ger. Pat. 281,877. See XIII.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Dyeing values of [natural Indian] dyestuffs.* J. P. Srivastava. Report to the Director of Industries of the United Provinces, India. J. Soc. Dyers and Col., 1915, 31, 122.

THE dyewoods containing red and yellow colouring matters used in Europe are of American origin; it is suggested that similar woods are also available in India. The dyeing values of thirteen colouring

matters have been investigated: Haldi (*Curcuma longa*), Cutch, (*Acacia catechu*), Safflower (*Carthamus tinctorius*), Lac dye, and Indigo are commercial products; Tun (*Cedrela Toona*), Tesu or Dhak (*Butea frondosa*), Arusa (*Adhoda Vatica*), and Jangli Nil or Wild Indigo (*Tephrosia purpurea*) are available but not commercial; Harsinghar (*Nyctanthes arbor tristis*) and Naspal (*Punica granatum*) require special arrangements for their collection; and the cultivation of Majith (*Rubia cordifolia*) and Sappanwood or Patang (*Caesalpinia sappan*) is believed to have practically ceased. Dye-trials on wool were carried out by immersion in an infusion of the colouring matter, alone, or with the addition of 1% of acetic acid; in some cases the dyed wool was treated with 2% bichromate, and in others wool previously mordanted with bichromate and oxalic acid or with aluminium sulphate and tartar emetic was used. Cotton was steeped overnight in myrobalon extract, squeezed, and then worked in fresh baths containing tartar emetic, stannous chloride, alum, or ferrous sulphate. The most brilliant results were obtained with stannous chloride, tartar emetic and alum being next in value; ferrous sulphate gave grey to black shades. Haldi (Turmeric) gave fairly fast dyeings on wool with the bichromate and oxalic acid mordant, but the colouring matter has the serious drawback that it is turned red by soap. Cutch (Katha), extracted from catechu wood, gives rise to Catechu Brown, one of the fastest colours known; cotton goods are boiled with extract of catechu to which has been added an amount of copper sulphate equal to one-tenth the weight of the colouring matter, the material is squeezed, allowed to stand a short time, boiled in a fresh hot bath containing 2% of potassium bichromate, and then washed and dried. Safflower (Kusum) is separated by macerating with water to extract the yellow colouring matter completely, and then extracting the pink colouring matter by means of dilute sodium carbonate solution; the pink colouring matter is valuable for dyeing cotton and silk; Safflower Yellow does not dye cotton on aluminium or tin mordant, but dyes wool direct. Lac dye yields beautiful scarlet and crimson shades on tin mordant. Harsinghar gives brilliant yellow shades with most mordants on wool; on wool mordanted with bichromate and oxalic acid previous to dyeing a beautiful brown is obtained; the dyeings on wool possess good fastness to milling with soap and soda. Tun gives the best shade on wool with the bichromate and oxalic acid mordant, but the dyeings are not very fast to milling with soap and soda. Arusa gave better results after precipitating the chlorophyll by addition of water to an alcoholic solution; the best shades on wool are obtained with the bichromate and oxalic acid mordant, the fastness of the dyeings being fair. Naspal (Pomegranate rind) gives yellow to full brown shades on wool, of very good fastness to milling. Tephrosia gives dull brown shades, of very good fastness to milling, on the various mordants; after purifying, by addition of water to the alcohol extract and washing out the chlorophyll with petrol, excellent yellow shades, comparatively fast to light, washing, and milling, were obtained. Majith dyes very fast shades on wool and cotton, the best results on cotton being obtained by the Turkey Red process; red, maroon, and claret shades of excellent fastness to light may be obtained with this dyestuff, which is identical with Madder. Sappanwood is similar to logwood, the oxidation of the colouring matter being carried out by the "aging" process; it can be used for the production of brilliant red, crimson, and purple shades, and is very suitable for calico printing.—F. W. A.

## PATENT.

*Electrolytic apparatus for the production of bleaching liquor, etc.* Eng. Pat. 1072. See XI.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Carbonic acid: Behaviour of — in pure aqueous and saline solutions.* E. Wilke. Chem. Ges. zu Heidelberg, Feb. 19, 1915. Chem.-Zeit., 1915, 39, 309.

The general assumption that aqueous solutions of carbon dioxide contain the hydrate,  $H_2CO_3$ , is not in accord with the probability that this substance being a hydroxyformic acid, would exhibit strong acidic properties. A re-determination of electrical conductivity of aqueous solutions of the gas, whilst confirming the values for the dissociation constant,  $2.89 \times 10^{-7}$ , previously found by Walker (Chem. Soc. Trans., 1900, 77, 1), demonstrated also the labile character of such solutions, changes in conductivity being effected by contact with metals, such as gold or platinum, and even by the feeble current used in making the measurements. The dissociation of carbonic acid in salt solutions was studied by determining the hydrogen-ion concentration, using a palladium capillary saturated from within with hydrogen under pressure, as electrode. Carbonic acid was found to be a much stronger acid under these circumstances than in pure aqueous solution, and the conclusion is drawn that in pure aqueous solution a higher hydrate, possibly ortho-carbonic acid,  $C(OH)_2$ , which is only a very feeble acid, is produced, from which by the dehydrating action of the salt solutions the lower hydrate,  $H_2CO_3$ , is formed, which possesses the strong acid properties expected of it. The fact that electrolysis of pure aqueous solutions of carbonic acid yields only minute traces of formic acid, compared with salt-containing solutions, is held to support this view.—G. F. M.

*Limestones for "fat" lime: Structure of —.* G. Gallo. Annali Chim. Appl., 1915, 3, 213—224.

In the quarrying of Carrara marble large quantities of small pieces are produced, and these represent a waste product, for the lime obtained on burning the marble, although containing over 97%  $CaO$ , is not a "fat" lime, and hence is unsuitable for building purposes, or for gas purification, sugar refining, manufacture of bleaching powder, or other purposes in which a condition of extreme fineness of subdivision is required. Experiments made by the author show that the quality of lime depends not only on the chemical composition, but also very largely on the structure of the limestone from which it is derived. In general a "fat" lime cannot be obtained from a limestone with a macrocrystalline structure, unless this be finely ground and then briquetted, before burning. In most cases grinding and briquetting are not practicable on account of the cost. To determine the quality of the lime the author used a test proposed by Giorgis and Cemi: 500 grms. of the lime is mixed with excess of water (about 1500 c.c.) in a tared cylinder of porous earthenware of about 2 litres capacity. The excess of water drains through the cylinder, and when the lime paste begins to show cracks on the surface, its volume is noted, and the cylinder with its contents is weighed. Under these conditions lime from Carrara marble absorbed about 920 grms. of water and yielded about 1.3 litres of lime paste, whereas a "fat" lime of similar composition from crypto-crystalline Capistrillo limestone absorbed 1960 grms. of water and yielded 2.3 litres of lime paste; lime from Carrara marble which was finely ground and passed through a sieve of 4900 meshes per sq. cm. before burning, absorbed 2060 grms. of water and yielded 2.3 litres of lime paste.—A. S.



*Lime; Reburning of*—*from alkali waste and other forms of precipitated carbonate of lime.* R. K. Meade. Met. and Chem. Eng., 1915, 13, 289—290.

LIME is recovered from waste calcium carbonate from the manufacture of caustic soda, sugar, etc., by settling or filtering the sludge and calcining in a rotary kiln as used in cement burning. The burnt lime consists of nodules of walnut size down to dust; it has a slightly yellowish or greenish colour and contains a small quantity of impurities taken up in the process; this is counteracted by adding 5—10% of fresh lime at regular intervals. The cost of a plant for treating 25 tons per day is stated to be \$17,000—20,000 (about £3500—£4200), with a fuel and power consumption of 600—800 lb. of coal or 60—80 galls. of oil and 15 kilowatt-hours per ton of lime produced. —W. R. S.

*Ammonia gas for inflating airships.* A. Sander. Chem.-Zeit., 1915, 39, 325—326.

A SUGGESTION has been made recently to substitute ammonia for hydrogen for aeronautical purposes, on the ground of its non-inflammability, its easy liquefaction whereby cylinders of liquid ammonia could be carried on board the airship for use as required, and its solubility in water whereby any desired degree of deflation could be attained by mere absorption in water. The author considers that the disadvantages attending the use of ammonia far outweigh these advantages. Its lifting power is only 0.52 kilo. per cub. metre compared with 1.20 kilos. for hydrogen, and volume for volume it is ten times as costly as hydrogen. Apart also from the destructive action of ammonia on the material of the envelope and its unpleasant effect on the occupants of the airship in case of leakage, mixtures of ammonia and air between the limits of 16.5 and 26.8%  $\text{NH}_3$  by vol. are dangerously explosive (see this J., 1914, 1087).—G. F. M.

*Hydrazine salts; Behaviour of certain—on decomposition by heat. [Detection of hydronitric (hydrazoic) acid in presence of sulphur dioxide.]* J. W. Turrentine. J. Amer. Chem. Soc., 1915, 37, 1105—1114.

HYDRAZINE monochlorate on decomposition in sulphuric acid solution, yields hydronitric (hydrazoic) acid. The corresponding di-salt gives the same result both in the presence and absence of sulphuric acid. The mono- and dichlorate of hydrazine yield no hydronitric acid in either acidified or neutral solution. Decomposition of the dry crystallised salt by heat in a stream of carbon dioxide produced hydronitric acid from the latter salt but not from the former; in both cases chlorine, nitrogen, and oxygen were liberated. Hydronitric acid can be detected in the presence of sulphur dioxide by boiling with sodium hydroxide and hydrogen peroxide until effervescence ceases, then acidifying, distilling, and testing the distillate with ferric chloride; a red coloration indicates hydronitric acid.—H. R. D.

*Hydrazine sulphate and ammonium hydrazide; Electrochemical oxidation of*— J. W. Turrentine and J. M. Olin. J. Amer. Chem. Soc., 1915, 37, 1114—1122.

THE investigation was undertaken to discover reactions that would yield nitrogen hydrides such as ammonia, and more particularly hydronitric (hydrazoic) acid as a product, under wider conditions than those obtained by Szarvasy (J. Chem. Soc., 1900, 77, 663). Hydrazine sulphate in saturated aqueous solution containing 10% by vol. of concentrated sulphuric acid is oxidised electrochemically to hydronitric acid with a

current of 1.5 amperes. The yield is greater when the electrolyte is maintained at 0° C. or lower by means of an ice bath. Ammonium hydroxide in the presence of sodium chloride and glue, using a current of 0.3—0.4 ampere, is oxidised to hydrazine provided that sodium chloride solution is added in small amounts at intervals during electrolysis.—H. R. D.

*Sodium and potassium; A new method for the determination of*—*in a mixture of their salts.* K. Okada. Mem. Coll. Sci., Kyoto Imp. Univ., 1914, 1, 89—93.

ONE GRM. of a mixture of sodium and potassium chlorides (or sulphates) and 2.4 grms. (or 2 grms.) of sodium hydrogen tartrate were dissolved in water and made up to 50 c.c. at 25° C. After shaking for 5 hours in a thermostat at 25° C. the residue from 20 c.c. of the clear supernatant liquid was ignited at a red heat and extracted with standard hydrochloric acid; the excess of acid was then determined with standard sodium hydroxide. From the amount of acid used the composition of the original mixture was determined by referring to a curve summarising the results obtained in the above manner with similar mixtures of known compositions. In a series of experiments with the chlorides the actual and determined molar percentages of potassium chloride were, respectively: 52.4, 52.3; 52.3, 52.3; 40.5, 39.5; 33.2, 33.0; 8.0, 8.3; 5.8, 6.6. The results obtained with sulphate mixtures were similarly: 91.1, 90.7; 84.9, 85.3; 79.3, 79.2; 50.6, 50.6; 43.2, 42.9; 25.9, 25.5; 2.8, 2.7. The method is unsatisfactory for chloride mixtures containing less than about 5% KCl; but with sulphate mixtures the degree of accuracy is not affected in this manner.—J. R.

*Sodium and potassium sulphates; The double salt of*— K. Okada. Mem. Coll. Sci., Kyoto Imp. Univ., 1914, 1, 95—103.

WATER was saturated with various mixtures of sodium and potassium sulphates, and from analyses of the solutions and residues (see preceding abstract) the existence of a double salt of the formula,  $\text{K}_2\text{Na}(\text{SO}_4)_2$ , was deduced. This salt forms solid solutions with sodium sulphate but not with potassium sulphate, and data are given for the interval 15° to 50° C.—J. R.

*Halides; Titration of small amounts of*— F. C. McLean and D. D. Van Slyke. J. Amer. Chem. Soc., 1915, 37, 1128—1131.

THE halide is precipitated in the presence of a known amount of free nitric acid (about 1 gm.) with an excess of N/25 or N/50 silver nitrate solution. In the case of bromides and chlorides the silver halide is removed by filtration. The excess of silver is titrated with N/50 potassium iodide which has been standardised against the silver nitrate solution. Before titrating, 4 c.c. of a solution containing 446 grms. of trisodium citrate, 19 grms. of sodium nitrite, and 2.5 grms. of soluble starch per litre is added for each gram of free nitric acid present. One drop of N/50 iodide produces a very sharp colour that can be seen in 150 c.c. of solution.—H. R. D.

*Sulphides; Oxidation of*—*with potassium iodate.* R. S. Dean. J. Amer. Chem. Soc., 1915, 37, 1134—1137.

ANDREWS (J. Amer. Chem. Soc., 1903, 25, 756) has shown that in solutions containing 50% or more of hydrochloric acid, potassium iodate may be used as an oxidising agent, the iodine first liberated being ultimately converted into iodine chloride, the disappearance of colour in some chloroform mixed with the liquid. In the determination of sulphides the method is found applicable, especially

where the sulphide is easily washed without oxidation, as in the case of As and Sb. In the oxidation of cadmium, zinc, and lead sulphides, one-third of the sulphur is oxidised to sulphuric acid, while two-thirds is liberated as free sulphur. (See also this J., 1911, 549; 1912, 411; 1915, 135.) —H. R. D.

*Calcium hydroxide and sulphur in aqueous solution: Reaction between*—(during preparation of lime-sulphur wash). S. J. M. Auld. Chem. Soc. Trans., 1915, 107, 480—495. (See van Slyke, this J., 1910, 589, and Tartar, this J., 1910, 837; 1914, 354.)

THE preparation and examination of lime-sulphur wash, under conditions which excluded air and carbon dioxide, indicated that lime and sulphur react in aqueous solution with the formation of polysulphides (or oxy-sulphides) and thio-sulphate only, probably according to the equations:  $5\text{Ca(OH)}_2 + 6\text{S} = 2(\text{CaS}_2\cdot\text{CaO}) + \text{CaS}_2\text{O}_3 + 5\text{H}_2\text{O}$ , and  $\text{CaS}_2\cdot\text{CaO} + x\text{S} + \text{H}_2\text{O} = \text{CaS}_{2+x} + \text{Ca(OH)}_2$ . When an excess of sulphur was used, the ratio between polysulphide S + monosulphide S and the monosulphide S alone, usually lay between 4.5 and 5.0, but sometimes exceeded the latter figure. Much of the polysulphide S was extracted by solvents, both from the solution and from the solid obtained by evaporation, and evidence was secured of the probable existence of a stable  $\text{CaS}_2$  complex; attempts to prepare calcium disulphide gave only Herschell's crystals, to which the formula,  $\text{CaS}_2\cdot\text{CaO}\cdot 7\text{H}_2\text{O}$ , is assigned (compare Gauthier, Annalen, 1884, 224, 178). It is suggested that the polysulphides of calcium possess the constitution  $\text{Ca} \left( \begin{smallmatrix} \text{S} \\ \text{S} \end{smallmatrix} \right)_x \text{S} : \text{S} : \text{S} : \dots$ , the atoms of sulphur in the chain becoming progressively more loosely attached (compare Küster and Heberlein, this J., 1905, 511). Experiments on the action of dilute acids upon the polysulphide solution indicated the possible existence of polysulphides at least as high as  $\text{CaS}_8$ . —F. SOPN.

*Barium oxide, acetic acid, water and magnesium oxide, acetic acid, water: The systems*—, J. Iwaki. Mem. Coll., Sci., Kyoto Imp. Univ., 1914, 4, 81—88.

A STUDY of the equilibrium of the systems at 25° C. established the existence of the following acid salts:  $3(\text{CH}_3\text{CO}_2)_2\text{Ba}\cdot 3\text{CH}_3\text{CO}_2\text{H}\cdot 11\text{H}_2\text{O}$ ;  $(\text{CH}_3\text{CO}_2)_2\text{Ba}\cdot 2\text{CH}_3\text{CO}_2\text{H}$ ;  $2(\text{CH}_3\text{CO}_2)_2\text{Mg}\cdot 3\text{CH}_3\text{CO}_2\text{H}\cdot 3\text{H}_2\text{O}$  and  $5(\text{CH}_3\text{CO}_2)_2\text{Mg}\cdot 10\text{CH}_3\text{CO}_2\text{H}\cdot 7\text{H}_2\text{O}$ . J. R.

*Cuprous salts of oxygen acids [cuprous oxalate], and a new method for preparing cuprous salts.* L. C. Daniels. J. Amer. Chem. Soc., 1915, 37, 1167—1171.

WHEN red cuprous sulphite ( $\text{Cu}_2\text{SO}_3\cdot\text{H}_2\text{O}$ ) is introduced in small quantities into an excess of a hot solution of oxalic acid, hydrated cuprous oxalate,  $\text{Cu}_2\text{C}_2\text{O}_4\cdot 2\text{H}_2\text{O}$ , separates as a brown powder. It is also formed by digesting 1 part of cuprous oxide with 24 parts of oxalic acid in solution. Cuprous oxalate dissolves in dilute hydrochloric acid (1:2), and in strong ammonia, without separation of copper; and it is decomposed by warm, dilute nitric and sulphuric acids, giving the cupric salt and free copper. Heated for 2 hours at about 95° C., its colour changes to a dull, dark gray, but the original colour is restored on subsequent treatment with cold dilute nitric acid. When heated in a crucible, it turns grey and then decomposes suddenly, almost explosively. —E. H. T.

*Tri-magnesium citrate.* E. Léger. Comptes rend., 1915, 160, 660—663. (See also this J., 1915, 574.)

THREE hydrated forms of tri-magnesium citrate

have been isolated containing respectively, 13, 9, and 7—8 mols.  $\text{H}_2\text{O}$ . The salt with 13 mols.  $\text{H}_2\text{O}$  crystallises out very slowly when a warm concentrated solution of citric acid is saturated with magnesium carbonate, and still more slowly when magnesium sulphate is added to tri-sodium citrate. The salt with 9 mols.  $\text{H}_2\text{O}$  is obtained as a crystalline precipitate when a strong solution of tri-magnesium citrate is kept on a water-bath for about 20 hours, or when the solution is simply heated to 110° C.; it is insoluble, even in boiling water, but dissolves in ammonia. The salt with 7—8 mols.  $\text{H}_2\text{O}$  is produced by mixing boiling alcoholic solutions of magnesium acetate (3 mols.) and citric acid (2 mols.); it dissolves in 2 parts of cold water, and the solution deposits crystals containing 13 mols.  $\text{H}_2\text{O}$  on standing. The latter compound loses 11 mols.  $\text{H}_2\text{O}$  at 110° C., and the remaining water is expelled at 150°—155° C. The salt with 9 mols.  $\text{H}_2\text{O}$  is more stable, complete desiccation not being effected below 175° C. Attempts to confirm the existence of basic magnesium citrates were unsuccessful. —E. H. T.

*Manganese dioxide: Preparation and transformations of colloidal*—, E. J. Witzemann. J. Amer. Chem. Soc., 1915, 37, 1079—1090.

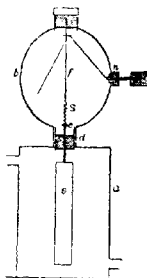
COLLOIDAL manganese dioxide is formed during the oxidation of certain organic compounds with potassium permanganate. A simple method of producing it is by the incomplete oxidation of dextrose with alkaline permanganate. 100 c.c. of potassium permanganate solution (60 grms. in 1100 c.c. water) is added from a dropping funnel, within a period of 5 minutes, to a solution of 5 grms. of dextrose in 20 c.c. of water, made alkaline with a few c.c. of 10% caustic soda. The sugar solution is placed in an ice-bath and kept in agitation. A viscous liquid is rapidly formed which in 5—10 minutes sets to a stiff jelly; this gradually changes to a semi-liquid gel, which in a few days forms a liquid dark-brown colloidal solution. The viscous liquid of the first stage has the properties of a typical emulsoid; the final solution seems to be of a suspensoid character. The conversion of the emulsoid into the jelly would be normal but for the slow but simultaneous conversion into the suspensoid, which is induced by the presence of the alkali. Both transformations are influenced by temperature changes, by changes in the concentration of the reacting liquids, and by the concentration of the alkali. The suspensoid is readily coagulated by warming, but otherwise is very stable, particularly in concentrated solution. Very similar results ensue if, instead of dextrose, galactose, levulose, lactose, tartaric acid, or glycerol be used, but lactose acts slowly, and in the case of the last two, the gels show little tendency to go into solution. It is regarded as probable that the stimulative effects of manganese in certain biochemical changes may be closely related to the existence and properties of the colloidal dioxide. —E. H. T.

*Mercuric sulphate: Action of the halogens upon*—, M. Chateau. Bull. Soc. Chim., 1915, 17, 121—124.

BRÜCKNER (Monats. Chem., 1906, 115) has shown that iodine reacts with mercuric sulphate under certain conditions to form a fairly stable complex compound,  $6(3\text{HgO}\cdot 2\text{SO}_3)\cdot 6\text{HgI}_2\cdot 11\text{HgI}_2\text{O}_6$ . The author has found that chlorine and bromine do not behave similarly, but that the products of their reaction upon mercuric sulphate are respectively sulphuric acid, hypochlorous acid, mercurous chloride, and sulphuric acid, hypobromous acid, mercurous bromide. —J. P. O.

*Radium; Practical methods for the determination of—*. I. *Interchangeable electroscopie and its use*. S. C. Lind. J. Ind. Eng. Chem., 1915, 7, 106—110.

A SIMPLE electroscopie (see fig.) for determining radium by the emanation method is described. The emanation chamber, *a*, consists of an upright brass cylinder, 4 ins. high and  $3\frac{1}{2}$  ins. diam., fitted with an inlet and an outlet tube and a collar screwed into the upper end plate. A brass cylinder, *c*,  $\frac{1}{2}$  in. diam., placed centrally in the cylinder and extending to within  $\frac{1}{2}$  in. of the bottom and top, serves as electrode. It is carried by a brass rod,  $\frac{1}{2}$  in. diam., which passes up through a plug of sealing wax, *d*, in the collar, and ends in a small conical cap, *e*, serving to make contact with the leaf system in the upper detachable part of the electroscopie. The latter consists of a horizontal cylinder, *b*,  $3\frac{1}{2}$  in. diam. and  $1\frac{1}{2}$  in. deep, the ends of which are closed by sheet mica held in place by steel wire rings fitting in grooves in a manner similar to that used for automobile tyres; fine wire gauze in contact with the inner face of the mica serves to lead off stray electrical charges. A telescope is fixed firmly to one end of the cylinder. The leaf system comprises a flat brass rod, *f*, terminating in a brass spring, *s*, to make a light contact with the cap, *e*; the aluminium leaf is attached to a small offset at the top of the brass rod by a moisture contact. The electroscopie is charged by means of a brass rod fixed in a hard rubber insulation, *u*, and sloping upwards inside the cylinder so as to make contact with the rod while charging, and then, by turning the rod, with the wire grating of the outer case. The electroscopie is charged for 15 mins., using just sufficient voltage to hold the leaf on the desired part of a micrometer scale on the eyepiece of the telescope, and the natural leak is then determined during 15 mins. The upper part is next detached, the lower chamber exhausted, and a dried mixture of emanation and air drawn in. After restoring normal pressure, the emanation is left in the chamber for 3 hours, the top replaced, and charged for 15 mins. as before. The time taken for the leaf to pass over a certain part of the scale is then noted; three readings are usually sufficient, but ten are taken if the deviations are greater than 1%. The instrument is calibrated by using the emanation evolved on dissolving a known weight (20—40 mgrms.) of high-grade pitchblende, the radium content of which is calculated from its uranium content. The quantity of emanation does not correspond exactly to the radium content, owing to the diffusion of a small fraction from the ore; the "emanating power" of the ore must be determined by sealing a weighed quantity in a tube, and, after 1 month or more, drawing off the emanation into an electroscopie by means of air. After each measurement dry, dust-free air from outside the laboratory should be drawn through the apparatus for some time, preferably over night. If the emanation chamber becomes contaminated with active deposit, it must be opened, the walls washed with dilute nitric acid (1:3), then with water, and dried, these operations being repeated until the natural leak of the instrument is sufficiently reduced.—A. S.



*Radium content of water from the Gulf of Mexico*. S. J. Lloyd. Amer. J. Sci., 1915, 39, 580—582.

A SAMPLE of water from the Gulf of Mexico gave a mean reading of  $1.70 \times 10^{-12}$  gram of radium per

litre. The measurements were made in the usual type of vacuum electroscopie with amber insulation, standardised with pitchblende containing 46.2% uranium; the mineral was dissolved in nitric acid, the solution diluted until 250 c.c. contained the same amount of radium as the sample of sea water, and kept for 30 days before using to ensure the presence of the maximum amount of emanation.—H. R. D.

*Kelps of the Pacific coast (U.S.A.); Organic constituents of—*. D. R. Hoagland. J. Agric. Research, 1915, 4, 39—58.

THE total organic matter in the seven species of kelp investigated, varied from 45 to 74% of the whole plant, and from 50 to 71% of the leaves and stems (dry basis). Most of it is insoluble in water and in alcohol, and some very complex compounds are present in a highly colloidal state. Pentosans were found in all samples, galactans in some, but starch and reducing sugars were absent. From one-third to one-fifth of the total nitrogen is water-soluble, and about the same proportion is non-protein nitrogen; the amounts of ammoniacal and amino-nitrogen were insignificant. Carbohydrates or similar substances compose the main portion of the organic matter, and are of colloidal character. Californian kelps yield no jelly similar to agar-agar. The most important carbohydrate present is "algin," which is best extracted with 2% sodium carbonate solution; it constitutes from 13 to 24% of the dry seaweed. Algin probably consists of a complex composed of compounds of the pentosan type and cellulose. It has weakly acid properties, and the sodium salt precipitates many metals from solution. Some "alginates" might be useful as sizes or mordants. The cellulose content composes about one-half of the crude fibre, or 3—4% of the whole dry plant. The total sulphur averaged about 1% of the dry plant, but *Fridaea* spp. contained 8.16%; nearly all of the sulphur is in organic combination. The iodine can be extracted by cold water or 90% alcohol, and is practically all in the inorganic state. The bromine content is only one-fifth to one-tenth that of the iodine, thus reversing the proportion, present in sea water. When the kelps are dried, much potassium chloride appears as an efflorescence, but this contains no iodine. It is very improbable that these kelps possess any value as nutriment for man or animals, and laboratory experiments indicated that the products of their destructive distillation are very much inferior to those from oak sawdust or fir shavings.—E. H. T.

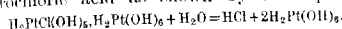
*Crystallisation from aqueous solutions; Velocity of—*. N. P. Campbell. Chem. Soc. Trans., 1915, 107, 475—480.

By comparing the rates of growth of weighed crystals of alum, suspended in a slightly supersaturated solution of this substance, it was found that the rates of growth on similar crystal faces were proportional to the areas of the faces exposed to the liquid. A large crystal did not grow at the expense of small ones. Alkaline impurities in the solution favoured the development of cube faces, and crystals of potassium alum, allowed to grow in a solution containing Portland cement, were obtained as almost perfect cubes, the octahedral faces being completely suppressed. In a solution saturated with regard to both alum and potassium bichromate, crystals of alum and bichromate, hung side by side, developed with perfect faces.—F. SODX.

*Electrolytes; Photochemical formation of—*. M. Boll. Ann. Phys., 1914, 2, 5—54, 226—268. J. Chem. Soc., 1915, 108, ii., 123—124.

THE photochemical decomposition of the chloroplatinic acids in very dilute aqueous solutions, on

exposure to the rays emitted by a mercury quartz lamp, was determined by measuring the increase in the electrical conductivity. A method of measuring high resistances was employed, involving the use of an alternating current and a quadrant electrometer. The decomposition of hexa-, penta-, tetra-, di-, and mono-chloroplatinic acids proceeded in agreement with the formula for a bimolecular reaction as represented in the following general equation:  $2\text{H}_2\text{PtCl}_6(\text{OH})_{6-n} + (2n-1)\text{H}_2\text{O} = (2n-1)\text{HCl} + \text{H}_2\text{PtCl}(\text{OH})_5, \text{H}_2\text{Pt}(\text{OH})_6$ . The solutions obtained were metastable, and usually deposited a reddish brown precipitate of platinum hydroxide, accompanied by a further rise in conductivity corresponding with the liberation of hydrochloric acid as shown by the equation:



Experiments with tetrachloroplatinic acid solutions subjected to monochromatic ultra-violet light ( $\lambda$  2536 and  $\lambda$  2655) showed that the reaction velocity is proportional to the intensity of the incident radiation, and absorption of the active rays is in accordance with the exponential formula representing the combination of Beer's and Lambert's laws. It is stated that the photochemical decomposition of the acids may be employed in a chemical actinometer ranging from the yellow to the extreme ultra-violet. From observations with monochromatic light (varying from  $\lambda$  2378 to  $\lambda$  5460) the reaction velocity is found to increase exponentially with the frequency, and for light of definite wave length the velocity increases considerably with rise of temperature. Experiments on the photochemical decomposition of oxalic acid in the presence of uranyl nitrate show that the reaction is unimolecular, as in the equation:  $\text{H}_2\text{C}_2\text{O}_4 + \text{O} \rightarrow 2\text{CO}_2 + \text{H}_2\text{O}$ , the uranyl nitrate acting as a catalyst. In agreement with this, the velocity is proportional to the quantity of light absorbed, and independent of the concentration of the oxalic acid.—B. N.

#### Indian salt-petre.

A PRESS *communiqué*, dated April 16, 1915, issued by the Indian Department of Commerce and Industry, states that, with a view to encourage the production of salt-petre and its allied salts in the areas in which the administration of the salt revenue is under the control of the Northern India Salt Revenue Department, the Government of India have recently sanctioned the adoption of the following measures: (1) A gradual extension of the areas in which the manufacture of salt-petre and its allied salts is permitted, provided that the extension is in no case allowed to imperil the security of the salt revenue; (2) a reduction of the licence-fees from Rs. 2 and Rs. 10 to R. 1 per annum in provinces other than Bihar; (3) distribution of the licences through the agency of the Post Office on payment of a commission of 2 annas to the postal clerk for each licence issued; (4) grant of permission to the owners of salt-petre refineries to compound for the duty on salt produced during the refining of salt-petre; and (5) removal of some of the restrictions to which factories for the manufacture of sulphate of soda (khari) are at present subject. The question of applying similar measures to the salt-petre industry in Bengal has also been referred for the consideration of the Government of Bengal. It is hoped these measures will remove the obstacles which at present hamper production and ensure an adequate supply of salt-petre and its allied salts to meet the requirements of local industries.

*Heat transmission capacity of a silica dish.* Lewis. See I.

*Reduction of iron oxides by platinum.* Sosman and Hostetter. See X.

*Determination of carbon dioxide in baking powder and carbonates.* Brubaker. See XIXa.

*Solubility of calcium and magnesium carbonates in waters free from carbonic acid in reference to their salt content.* Gothe. See XIXb.

*Colorimetric detection of oxalic acid and of manganese.* Sachler. See XXIII.

*Precipitant for ammonia.* (Substitute for Nessler's reagent.) Graves. See XXIII.

#### PATENTS.

*Acid-spray catcher.* F. Tschudy, Fairfield, Ala. U.S. Pat. 1,135,171, April 13, 1915. Date of appl., Sept. 24, 1914.

The apparatus comprises an outer and an inner shell, with the intermediate space divided into two compartments by division walls; baffles-plates are placed alternately on the inner and outer walls. The space enclosed by the inner shell forms an outlet conduit, which is divided by a partition into two compartments communicating respectively with the corresponding outer compartments. Means are provided for directing the gas carrying the acid spray to one of the outer compartments and shutting off the other, whereby the latter may be washed without interrupting the flow of gas.—H. R. D.

*Nitric acid: Process and apparatus for the preparation of highly concentrated*——. R. Schall, and Stickstoffwerke A.-G. Ger. Pat. 280,965, Aug. 30, 1913.

A MIXTURE of dilute nitric acid and a dehydrating agent is passed through a tower provided with a central tube through which heating gases are passed. Ribs of heat-conducting material, projecting from the outer wall of the tube, are arranged in steps or in the form of a screw so as to lengthen the path of the acid along the surface of the tube. The tube and ribs are preferably made of acid-resistant iron-silicon or iron-chromium alloys.—A. S.

*Sulphuric acid: Apparatus for concentrating*——. A. Bernutat. Ger. Pat. 281,133, March 12, 1914.

The apparatus comprises three concentrating compartments arranged in series, through which the acid flows in succession, meeting hot gases flowing in the opposite direction. Special conduits are provided through which a portion of the acid in the second and third compartments can flow back to the first, thereby raising the concentration and temperature of the acid in the latter.—A. S.

*Sulphuric acid: Reaction tower system for the manufacture of*——. E. Hoedling. Ger. Pat. 281,135, Dec. 24, 1912.

The towers are packed with tubes provided with openings and partitions so as to afford a large number of reaction spaces without unobstructed passages.—A. S.

*Ammonia and formic acid: Preparation of*——. H. Haack. Ger. Pat. 281,044, Feb. 11, 1913.

AMMONIA and formic acid are produced by heating compounds of the type of ferrocyanides with water or bases. With potassium ferrocyanide the reaction proceeds according to the equation:  $\text{K}_4\text{Fe}(\text{CN})_6 + 14\text{H}_2\text{O} \rightarrow \text{HCO}_2\text{K} + 2\text{HCO}_2\text{NH}_4 + 4\text{NH}_3 + \text{Fe}(\text{OH})_2$ .

If decomposition be effected with potassium hydroxide solution, a pure solution of potassium formate is obtained after distilling off the ammonia and separating the precipitated iron hydroxide.—A. S.

*Ammonia from gases, vapours, and spray; Process for the recovery of*—A. G. der Chem. Produktenfabr. Pommernsdorf, and R. Siegler. Ger. Pat. 281,095, April 29, 1913.

THE gas, vapour, or spray is mixed with sulphur dioxide, and ammonium sulphite is separated in the form of a concentrated aqueous solution by mechanical treatment of the mixture in a warm atmosphere saturated with moisture, cooling being avoided. For instance, the mixture may be drawn into the casing of a centrifugal fan. The gases leaving the fan casing still contain small quantities of ammonia and sulphur dioxide, which are recovered in the form of a concentrated solution of ammonium sulphate by passing the gases through a wooden tower packed with wood charcoal or coke. Hydrochloric acid may be used instead of sulphur dioxide.—A. S.

*Ammonia from coke breeze; Process for obtaining*—C. Waldeck. Ger. Pat. 281,096, Aug. 15, 1913.

THE coke breeze is treated with steam or a mixture of steam and other gases at the temperature most favourable to the conversion of its nitrogen into ammonia, the necessary temperature being attained by passing an electric current through the coke. From 88 to 99% of the nitrogen present in the coke can be converted into ammonia.—A. S.

*Ammonia; Preparation of— from a mixture of carbon monoxide, nitrogen, and water.* J. Wolf. Ger. Pat. 281,317, Dec. 11, 1912.

A GASEOUS mixture rich in carbon monoxide and nitrogen is produced, for instance by leading air over incandescent coke or the like, and after leaving the generator, is mixed with steam or atomised water. Ammonia, ammonium salts or derivatives, and carbon dioxide are formed, and the yield can be increased by working under pressure: the temperature should be kept between 300° and 450° C.—A. S.

*Hydrocyanic acid; Preparation of— synthetically by the circulating passage of a gas mixture through an expanded high potential arc.* A. V. Lipinski, Zürich, Switzerland. Eng. Pat. 11,107, May 5, 1914.

A GASEOUS mixture of hydrocarbons, hydrogen, and nitrogen is circulated through an expanded high-potential arc, in which separation of carbon is prevented by blowing in cooled residual gases (freed from hydrocyanic acid) directly behind the point of highest temperature of the reaction mixture. A portion of the residual gas is burnt with air in a special combustion chamber, in order to eliminate hydrogen and replace the nitrogen used in the reaction, and oil gas or natural gas is then added and the mixture again passed into circulation. The yield of hydrocyanic acid may be increased by carburetting the oil gas or natural gas with paraffin hydrocarbons.—F. SODX.

*Hydrogen cyanide and cyanogen compounds; Preparation of— by catalytic combination of gaseous or volatile compounds of carbon and nitrogen.* C. Beindl. Ger. Pat. 281,723, May 14, 1914.

HIGHER yields are obtained by mixing compounds of the alkalis, alkaline-earth, or earth metals or of molybdenum, uranium, osmium, titanium, etc., or the corresponding ores, with the metal or metals used as catalyst.—A. S.

*Alkali lyes; Process for the dehydration of*—W. Hentschel. Ger. Pat. 281,792, Aug. 30, 1912.

ALKALI lyes are evaporated by passing them through a series of iron retorts, the solution flowing

as a film over the horizontal bottom of each retort. Concentration to 96–99% strength is effected in retorts heated to a temperature below red heat, and the last traces of water are expelled in retorts heated to redness. Caustic alkali containing 1–1% H<sub>2</sub>O vigorously attacks red-hot iron, but has little action on iron at lower temperatures; alkali containing not more than 1% H<sub>2</sub>O has little action on red-hot iron.—A. S.

*Calcium carbide; Manufacture of*—F. M. Becket, Niagara Falls, Assignor to Union Carbide Co., New York. U.S. Pat. 1,137,567, April 27, 1915. Date of appl., Jan. 20, 1915.

A MIXTURE of lime and bituminous coal, in such proportion that the fixed carbon approximately suffices for the carbide-forming reaction, is coked in a by-product oven, so as to yield a mechanically strong aggregate, in which electrodes may be embedded for smelting to carbide.—F. SODX.

*Alumina; Method of making alkali-soluble*—P. R. Hershman, Chicago, Ill., Assignor to The Mineral Products Co., New York. U.S. Pat. 1,137,617, April 27, 1915. Date of appl., Oct. 29, 1914.

ALUMINA, e.g., calcined alunite, is heated with carbon, in presence of a protective (non-nitrifying) gas, to a temperature sufficient to leave it soluble in alkali, mainly as oxide.—F. SODX.

*Hydroxides of metals; Production of— by means of ammonia.* H. A. Frasch, New York. U.S. Pat. 1,135,785, April 13, 1915. Date of appl., April 10, 1913.

A SOLUTION of a salt of the metal, e.g., nickel, is boiled in the presence of an ammonium salt and free ammonia, until the hydrated oxide is precipitated. The aqueous ammoniacal vapour evaporated during the process is condensed, and sufficient of the distillate returned to keep the boiling solution alkaline with ammonia during the precipitation of the metallic oxide; or the vaporised ammonia may be concentrated by means of a dephlegmator, and a part of the dehydrated ammonia returned to keep the boiling solution alkaline.—H. R. D.

*Kelp; Extraction of mineral salts and organic constituents of*—A. G. Artz, Assignor to Kelp Products Co., San Francisco, Cal. U.S. Pat. 1,136,390, April 20, 1915. Date of appl., March 10, 1913.

RAW kelp is subjected to pressure, sulphuric acid is added to the expressed liquid to precipitate the algin, and after filtration, calcium carbonate is added in sufficient quantity to neutralise the acid, and the liquid is made alkaline by the addition of sodium carbonate (10 to 15 lb. per 100 gallons of liquid). The solid organic matter is macerated to a pulp and boiled with the alkaline solution obtained, to extract further algin and the soluble salts contained in the pulp. The solution thus obtained is separated from the cellulose by pressure, and treated with acid to precipitate dissolved algin, filtered, neutralised, and evaporated to remove the salts.—H. R. D.

*Potash salts and the like; Apparatus for the continuous decomposition and dissolving of*—G. Sauerbrey Maschinenfabr. A.G. Ger. Pats. (A) 280,557, March 21, 1914, (B) 281,354, Sept. 21, 1913, (C) 281,355, March 21, 1914, and (D) 281,356, Sept. 21, 1913. Additions to Ger. Pat. 262,235.

(A). THE ends of the inner drum of the apparatus described in the chief patent (this J., 1913, 826) are in the form of double cones projecting through the ends of the fixed outer drum and supported outside the latter, on rollers. The driving wheel

for rotating the inner drum is also outside the fixed outer drum, and hence the bearings, etc., are accessible while the apparatus is working. (B). The lower part of the fixed outer drum is enlarged to form a trough in which is placed a screw. The screw serves to keep the fine solid material in suspension until it is dissolved and to carry forward the heavier material to an elevator which returns it to the inner drum. (C) The solution from the inner drum carrying suspended particles passes first into a heating chamber below the outer drum. This chamber has steeply inclined side walls and is provided with heating pipes and a screw conveyor and agitator at the bottom. It is connected with the outer drum by a series of conduits, between which are transverse partitions so as to cause the liquid to circulate upwards and downwards. Pieces of angle iron, fixed on the outer surface of the inner rotating drum, between the conduits connecting the outer drum and the heating chamber, are so arranged as to cause the solution to circulate from the middle towards the ends. (D) The apparatus consists of two V-shaped troughs, one within, but spaced apart from the other. The material is disintegrated and partly dissolved in the inner trough provided with a screw conveyor and agitator, and flows thence through an opening at the middle into the outer trough, in the lower part of which is another screw conveyor. The inner trough is provided with transverse partitions above the conveyor, and in the spaces between the partitions are agitating blades mounted on a shaft. The liquid carrying suspended matter rises in the outer trough and is caused by baffles to take a sinuous path around heating tubes in the spaces between the side walls of the two troughs. —A. S.

*Potassium chloride in a coarsely crystalline form: Process for obtaining* ——. Consolidierte Alkali-Werke A.-G. f. Bergbau u. Chem. Industrie. Ger. Pat. 281,501, July 2, 1907.

A COOLING liquid is passed through cooling boxes arranged side by side in a trough, and the hot solution of potassium chloride is passed through the trough over the outer surfaces of the cooling boxes, in the opposite direction to that of the cooling liquid. The crystals deposited on the outer walls of the cooling boxes fall to the bottom of the trough when the deposit attains a certain thickness, and are removed by a conveyor. —A. S.

*Crystals: Method of removing from the crystallisation plates of apparatus for crystallising hot-saturated solutions of salts.* G. Saurebrey Maschinenfabr. A.-G. Ger. Pat. 280,915, Aug. 30, 1913. Addition to Ger. Pat. 271,216 (this J., 1911, 184).

THE crystals are removed by a powerful jet of a mother-liquor or other solution incapable of dissolving them. —A. S.

*Albite and the like: Treatment of* ——. C. H. MacDowell, Chicago, Ill., Assignor to The Mineral Products Co., New York. U.S. Pat. 1,136,549, April 20, 1915. Date of appl. July 31, 1914.

ALBITE is calcined to drive off sulphurous and sulphuric anhydrides, then heated to a higher temperature in the presence of carbon, and preferably in a current of gas, in order to volatilise and drive off the potash, which is deposited as a solid. —H. R. D.

*Radioactive constituents from liquids containing them: Process for obtaining* ——. H. Stern. Ger. Pat. 280,694, Jan. 10, 1914.

THE radioactive liquid is passed through a filter composed of substances, e.g., natural or artificial zeolites, capable of exchanging bases with the radioactive substances. —A. S.

*Chlorides, especially stannic and titanous chlorides: Process for reducing anhydrous, volatile* ——. F. Meyer and H. Kerstein. Ger. Pat. 281,091, April 30, 1913.

THE chloride is reduced by heating it in admixture with a current of hydrogen or equivalent gas. The gases leaving the reaction chamber are cooled somewhat to condense the reduction product or products and then returned to the reaction chamber in the opposite direction. If an intermediate product is formed, condensation is effected in stages, and the intermediate product is caused to pass in counter-current to the pure final product, free from reducing agent, on its way back to the reaction chamber. The process is applicable to the reduction of the stannic chloride obtained as a by-product in tin-plating, and to the preparation of metals, such as titanium, not easily obtained in a pure condition by other methods. (See also this J., 1913, 1915.) —A. S.

*Boric acid compounds [pyretetricharates] containing active oxygen: Preparation of* ——. J. Auer. Ger. Pat. 281,134, July 4, 1913.

PERITETRABORIC acid or its salts are prepared by the interaction of tetraboric acid (pyroboric acid) with hydrogen peroxide or a mixture of hydrogen peroxide and a metallic peroxide at not above 15° C. and preferably below 0° C. The sodium salt has the formula  $\text{Na}_2\text{B}_4\text{O}_{11}$ . —A. S.

*Oxygen and nitrogen: Preparation of active* ——. O. Bender. Ger. Pat. 280,966, Feb. 16, 1913.

WATER which has been treated with nitrogen and oxygen under pressure is delivered continuously to the porous walls of a closed, heated generator. The mixture of active oxygen and nitrogen thus produced forms ammonium nitrite when brought in contact with water. It may be used for sterilising water and for converting sulphur dioxide into sulphuric anhydride or acid. —A. S.

*Oxides of nitrogen: Preparation of* ——. by combustion of air with the aid of carbon compounds. E. Herman. Ger. Pat. 281,081, April 24, 1913.

METHANE is burnt with air enriched in oxygen in a chamber maintained under high pressure and connected with a condenser in such a manner that condensed water cannot flow back into the reaction chamber. Combustion may be effected in the ordinary way with excess of oxygen, or the methane may be burnt by flameless surface combustion within a mass of zircon. A yield of 3–12% by vol. of oxides of nitrogen is obtained, with a consumption of about 2.5 cub. m. of methane per kilo. of 100%  $\text{HNO}_3$ . —A. S.

*Carbon monoxide from oxygen and carbon: Furnace for the manufacture of pure* ——. Ges. f. Chem. Ind. in Basel. Ger. Pat. 280,968, Sept. 25, 1913.

THE furnace is cooled by means of a water-jacket at and near the combustion zone. The oxygen is introduced through a narrow, water-cooled iron pipe, terminating in a nozzle, which projects upwards through the centre of the base of the furnace into the charge of carbon. —A. S.

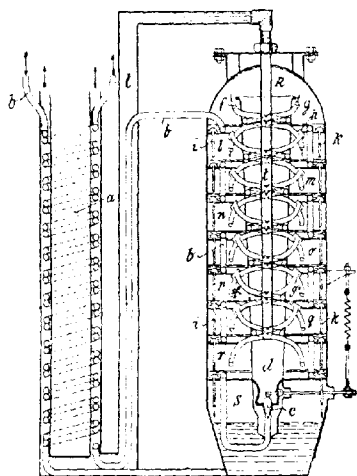
*Carbon dioxide from alkaline-earth carbonates: Apparatus for obtaining* ——. F. Kopp and R. Goerg. Ger. Pat. 281,132, May 4, 1913.

THE material is carried through the calcining chamber on a travelling support, in order to avoid formation of dust. —A. S.

*Liquid air: Apparatus for producing and separating it into its constituents.* A. Neumann. Ger. Pat. 281,518, March 2, 1913.

COMPRESSED air passes through the coil, b, and valve, c, into the tapering tube, d, and is gradually

liquefied. When the tube, *d*, is filled, the liquid air overflows into *R*, and flows down through the compartments, *l*, *m*, *n*, *o*, *p*, *q*, *r*, by way of the



tubes, *l*, *k*. The liquid is fractionated in these compartments, and the evolved gas flows into the tubes, *f*, *g*, which pass through the tube, *d*. In these tubes the gas is partially condensed, and the condensed liquid flows into the next lower compartment, whilst the gas rich in nitrogen, which is prevented from returning to the compartments, *l*, *m*, etc., by hoods, *h*, passes through connecting tubes to the tube, *t*, and flows away through the heat-exchanger, *a*. The liquid becomes enriched in oxygen as it descends, and practically pure liquid oxygen collects in *s*.—A. S.

*Cyanides: Process for producing*—J. E. Bucher, Providence, R.I., U.S.A. Eng. Pat. 13,332, May 30, 1914. Under Int. Conv., Nov. 7, 1913.

See U.S. Pat. 1,120,682 of 1914; this J., 1915, 176.

*Carbonitrides: Process of producing*—S. Peacock, Chicago, Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, U.S. Pat. 1,137,524, April 27, 1915. Date of appl., Feb. 11, 1912.

See Eng. Pat. 11,390 of 1912 and Fr. Pat. 458,168; this J., 1913, 699, 1937.

*Hydrogen peroxide: Process of manufacturing*—W. Weber, Assignor to Henkel and Co., Düsseldorf, Germany, U.S. Pat. 1,138,519 and 1,138,520, May 4, 1915. Date of appl., Feb. 26, 1914.

See Eng. Pat. 754 of 1914; this J., 1914, 644.

*Process for effecting chemical reactions at high temperatures*. Ger. Pat. 281,004. See I.

*Electrolytic apparatus for the production of bleaching liquor, etc.* Eng. Pat. 1672. See XI.

## VIII.—GLASS; CERAMICS.

*Stained glass: Contribution to the chemical study of medieval*—G. Cheneau. Comptes rend., 1915, 160, 622–624.

The following analytical results were yielded by fragments of stained glass of various colours, dating

from the end of the 13th century, which were detached from the large west rose-window of Rheims Cathedral by a storm in 1886. The data in the fifth column refer to a specimen of the same period from an unknown source.

	Violet.	Blue.	Green.	Red.	Red (L. Appert)
SiO <sub>2</sub> .....	54.30	53.90	48.60	55.50	56.25
TiO <sub>2</sub> .....	0.2	0.20	trace	trace	—
SO <sub>2</sub> .....	—	0.20	—	—	—
Loss on ignition .....	0.30	0.40	0.30	0.40	—
Al <sub>2</sub> O <sub>3</sub> .....	4.20	3.90	5.70	3.00	8.15
CaO .....	12.60	19.30	14.90	17.80	14.35
MgO .....	4.70	4.10	5.50	6.10	—
K <sub>2</sub> O .....	18.70	12.20	18.10	15.00	17.30
Na <sub>2</sub> O .....	1.30	1.90	1.10	1.80	—
PbO .....	—	trace	—	0.03	—
Bi <sub>2</sub> O <sub>3</sub> .....	—	0.62	—	—	—
CuO .....	0.02	0.13	1.81	—	not state
CoO .....	trace	0.25	0.06	—	—
NiO .....	0.06	—	trace	—	—
Mn <sub>2</sub> O <sub>4</sub> .....	1.85	3.03	2.39	0.86	—
Fe <sub>2</sub> O <sub>3</sub> .....	1.20	0.79	1.65	0.84	3.00
	99.43	100.32	100.11	99.46	99.05

The results illustrate the common origin of the Rheims specimens, which was evidently different from that of the specimen examined by Appert. The violet glass contains manganese as Mn<sub>2</sub>O<sub>4</sub> associated with the metallic oxides which occur in impure pyrolusite, and to which the flesh-coloured tinge of 13th century violet glass is attributed. The blue specimen contains the elements occurring in native cobalt sulpharsenide, with the exceptions that the nickel has been eliminated and the copper and manganese reinforced. The red colour is due to cuprous oxide, and the green to the usual copper-iron mixture, containing also a little cobalt and much manganese. The high potash content of the violet and green specimens is attributed to the addition of saltpetre for the purpose of overcoming the reducing action of the furnace gases.—J. R.

## PATENTS.

*Glass furnace*. E. Miller, Columbus, Ohio, U.S. Pat. 1,135,973, April 13, 1915. Date of appl., Feb. 19, 1912.

THE working chamber is an annular trough, in which the molten glass is given a circular motion, past the working hole, by means of a flanged device which can be rotated and dips just below the surface of the glass. —W. C. H.

*Glass and art of making the same*. H. M. Brookfield, New York, U.S. Pat. 1,136,504, April 20, 1915. Date of appl., Jan. 27, 1912.

A NON-CRYSTALLINE, glassy compound is made by heating a mixture of about 100 lb. of pulverised glass or cullet with 30 lb. of pulverised clay, with or without addition of lime, to about 2500 F. (1370° C.), and cooling and annealing the product. —W. C. H.

*Muffle for the distillation of zinc*. U.S. Pat. 1,136,519. See X.

## IX.—BUILDING MATERIALS.

*Portland cement: Value of the high-pressure steam test of*—R. J. Wig and H. A. Davis. Technol. Paper (U.S. Bureau of Standards), No. 17. J. Franklin Inst., 1915, 179, 597–599.

CEMENT pats and briquettes, after 24 hours storage in a damp closet, were treated in steam at 300 lb. per sq. in. pressure for an hour, the total

time in the boiler being 3 hours. Some cements, mixed neat, which are sound according to the standard atmospheric steam test, but unsound in the high-pressure steam, in time show signs of unsoundness when stored under normal conditions in dry air. There is no difference in the cementing quality of cements, and hardly any in the linear change of prisms made of cements, which pass or are rejected by the high-pressure steam test. About 70% of the brands examined passed the high-pressure test. Apparently the coarse particles of a normally unsound cement cause the expansive action, and after ageing for 2 to 6 months such a cement will usually pass the high-pressure test. It is recommended that the test should be made on all cements or concretes that are to be cured in steam above atmospheric pressure. It may forecast the behaviour of cement when exposed in dry air, but not that of concrete as normally exposed, and does not indicate superior cementing power, as judged by the compressive strength. Cements which pass the test do not make more permanent or durable concrete than those which fail to pass it. In some cases, cements which fail to pass the standard atmospheric steam test, but are satisfactory otherwise, may show normal strength in concrete. For practical constructional work, the high-pressure steam test apparently does not forecast the ultimate soundness of concrete. W. C. H.

*Portland cement: British standard specification for* —. Engineering Standards Committee, 28, Victoria Street, London, S.W. Price 5s.

In the new edition of the British Standard Specification for Portland cement, increased fineness of grinding of cement has been legislated for, and the minimum tensile strength at seven days of both neat cement and cement and sand has been raised. The clauses dealing with the preparation of the briquettes have been amplified, the procedure being described in greater detail than has previously been the case. In the case of both neat cement briquettes and cement and sand briquettes, ramming or hammering is prohibited. The increase of tensile strength at twenty-eight days both for neat cement and cement and sand is now given as a formula in place of the fixed percentages which have been specified hitherto.

*Refractories: Thermal conductivity of* —. R. Dudley, jun. Amer. Electrochem. Soc., April, 1915. Met. and Chem. Eng., 1915, 13, 315-316.

To determine the thermal conductivity of refractory brick under conditions approaching those of actual practice, a furnace having three permanent walls was used, the fourth being constructed with the bricks to be tested (fireclay, silica with clay bond, silica with lime bond, and magnesite). The mean conductivities found, expressed in gram-calories per second per cub. in. per 1° C., were:—

	At 100° C.	At 1000° C.
Fireclay .....	0.0043	0.0086
Silica (clay bond) .....	0.0051	0.0086
Silica (lime bond) .....	0.0056	0.0108
Magnesite at 445°-830° C. ....	0.0113.	

W. R. S.

*Fibron [silicon oxycarbide], a new heat insulator.* E. W. Weintraub, Amer. Electrochem. Soc., April, 1915. Met. and Chem. Eng., 1915, 13, 315.

FIBRON, a silicon oxycarbide, is prepared by melting silicon with a catalytic agent, such as calcium fluoride, in a muffle or crucible, carbon monoxide and dioxide diffusing slowly through the containing vessel and combining with the silicon

to a soft, resilient, fibrous material. The average diameter of the fibres is 0.6  $\mu$ ; the specific gravity is 1.8-2.2, while the apparent density is 0.0025-0.0030; the volume occupied by air is therefore 99.5-99.9%. The heat insulating capacity is equal to that of compressed wool of fifteen times greater density.—W. R. S.

*Structure of limestones for "fat" lime.* Gallo. See VII.

#### PATENTS.

*Wood preserving medium.* Grubenholzimpregnierung Ges. Ger. Pat. 281,691, Oct. 28, 1913.

POLYXINTROPHENOLS and their nuclear substitution products in which the hydrogen atom of the hydroxyl is replaced by an alkyl or acyl group, are used as wood preservatives. Acetyldinitrophenol, m. pt. 64° C., is specially suitable. It is easily soluble in organic solvents with the exception of petroleum spirit.—A. S.

*Slurry: Apparatus for mixing* —. A. Schröder, Charlottenlund, Denmark, Assignor to F. L. Smith and Co., New York. U.S. Pat. 1,137,294, April 27, 1915. Date of appl. Feb. 21, 1914.

THE mixing tank is provided with a number of nozzles projecting through the sides and bottom, for blowing air through the charge in horizontal and vertical directions, and with one or more mechanical agitators having horizontal, hollow arms from which horizontal streams of air are discharged during rotation.—W. E. P. P.

*Disinfecting and preserving agent.* Ger. Pat. 281,812. See XIXb.

*Apparatus for heating, drying, and pulverising earthy materials [for paving].* Eng. Pat. 9281. See I.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Open-hearth furnace: Heating up — by means of tar.* A. Greiner, Iron and Steel Inst., May, 1915. [Advance proof.] 1 page.

AT the Cockerill works (Liège), owing to the shortage of coal due to the war, a 12-ton open-hearth furnace was heated for 10 weeks with coke-oven tar, producing 1896 tons of steel in 215 charges, with an average consumption of 300 lb. of tar per ton, including the amounts required for lighting up and for heating during stoppages; in uninterrupted working the average consumption of tar is 250 lb. per ton. The tar was delivered to the burners at a pressure of half an atmosphere and sprayed with air at a pressure of 3 atmospheres. A new 25-ton furnace, running exclusively on tar, has been in operation for several weeks.—W. R. S.

*Iron: Nature of the A2 transformation in* —. K. Honda, Iron and Steel Inst., May, 1915. [Advance proof.] 43 pages. (See also this J., 1914, 83, 595.)

THE present investigation is based on the magnetism, thermal expansion, electrical resistance, and other properties of iron, the results confirming the author's previous view that the A2 transformation is not an allotropic change but an intermolecular alteration occurring gradually in the  $\alpha$  phase during a large interval of temperature; from the standpoint of the phase rule,  $\delta$ -iron has no existence, being only a condition of  $\alpha$ -iron between the critical and A3 points. The magnetic changes observable in nickel, cobalt, and other ferro-magnetic materials at their respective critical ranges are ascribed to a similar cause. According to the theory of ferro-magnetism advocated, the



molecules of ferro-magnetic substances are nearly spherical and those of paramagnetic bodies flattened or elongated. The A2 transformation, or that of any ferro-magnetic into a paramagnetic body at high temperature, is regarded as the consequence of the gradual deformation of the spherical molecules with rise of temperature, the heat evolved or absorbed being the energy of deformation and that imparted to the molecules to cause their rotatory vibrations.—W. E. F. P.

*Iron; The diffusion of carbon in*—. E. W. Adams. Iron and Steel Inst., May, 1915. [Advance proof.] 14 pages.

HIGHLY polished specimens of steels of different carbon content were bound together and heated in a vacuum to 900–1000° C. Welding took place at the points of closest contact, with diffusion of carbon from the high-carbon to the low-carbon specimen. When the specimens were not in contact both lost weight. Specimens not in contact, when heated in a rarefied atmosphere of carbon monoxide showed a slight transference of carbon from the high-carbon to the low-carbon specimen. Highly polished specimens of the same steel bound together and heated in a vacuum, also welded together at the points of closest contact. The amount of welding or intercrystallisation taking place between two samples of iron or steel was found to be independent of the carbon-content or the difference of carbon-content of the two specimens, and dependent only on the closeness of contact between the two surfaces.—T. St.

*Iron, carbon, and phosphorus*. J. E. Stead. Iron and Steel Inst., May, 1915. [Advance proof.] 41 pages. (See also this J., 1914, 173.)

A SUMMARY of previous work is given and the results of recent investigations are discussed more fully. When iron containing 2% P and 0.12% C is carburised by cementation, the ternary eutectoid  $\text{Fe}_3\text{P}-\text{Fe}-\text{Fe}_3\text{C}$  (closely resembling pearlite) is formed, and it was found that, by subjecting this phosphorised iron to an annealing process such as is used in making malleable iron, with the object of removing carbon and causing diffusion of phosphorus from the eutectic into the iron, crystalline plates of pure  $\text{Fe}_3\text{P}$  were formed along the cleavages of the iron crystals. With increasing carbon, phosphorus becomes more and more concentrated in the portions solidifying last; the longer the period of cooling, the greater is the tendency to form local, intercrystalline segregations which become "ghost lines" on being extended by forging. Unless associated with a material quantity of slag inclusions, the presence of ghost lines does not appear to be injurious to the strength of steel. Experiments are described showing the white ghost lines in a soft steel plate (0.06% P) to be the parts rich in phosphorus, the last to be invaded by carbon on heating, and the first to part with carbon on cooling; and the parts of iron or steel bars containing phosphorus concentrations to be the most resistant to carburisation by cementation. A new reagent and method (used throughout the present investigation) for detecting variations in the phosphorus content of iron and steel is also described. The reagent is prepared by dissolving 10 grms. of cupric chloride and 40 of magnesium chloride in a little hot water, adding 20 c.c. of hydrochloric acid, and diluting the solution to 1000 c.c. with alcohol. The surface of the specimen is covered with a thin layer of the liquid (not immersed) which is shaken off after 1 min. and replaced by another layer, the process being repeated as often as necessary; after washing with boiling water and methylated spirit in quick succession, the spirit is quickly evaporated by shaking the heated specimen. By this means, copper is deposited, as a hard, coherent layer, first

on the portions containing least phosphorus, but eventually (by repeated applications) over the whole surface of the specimen, the selective action of the reagent being sensitive to differences of less than 0.01% P. The method gives only differential, not absolute results, the rate of attack increasing as the phosphorus content of the metal decreases; with this reagent elements other than phosphorus (e.g., Ni, Cu, As, Cr, Sn, Sb), existing in a state of variable solid solution (i.e., unevenly distributed) in alloys, may also give similar indications.—W. E. F. P.

*Steel; Detection of burning in*—. J. E. Stead. Iron and Steel Inst., May, 1915. [Advance proof.] 5 pages.

"BURNING" in steel is the result of heating the metal to incipient fusion, and is not due to the formation of films of oxide by the passage into the steel of the oxidising gases of the furnace. The oxide found between the crystals is formed after the material has broken up, when air readily enters the open fissures. The first liquid that forms at the temperature of incipient fusion is rich in phosphorus, and the presence of phosphorus-rich globules and crystal-envelopes is the criterion of "burning." Metal which is simply redshort resembles burnt steel in having cracks lined with oxide, but is free from the globular specks rich in phosphorus. Portions free from cracks should therefore be selected for polishing and etching in investigations as to "burning." The best etching reagent for revealing the phosphorus-rich globules and envelopes is that described in the preceding abstract.—T. St.

*Iron, silicon, and carbon alloys; Researches on the*—. G. Charpy and A. Cornu-Thenard. Iron and Steel Inst., May, 1915. [Advance proof.] 27 pages.

THE transformation points were investigated by thermal and dilatation measurements in dead soft steels (C 0.10–0.15%; Si 0.1–7%), semi-hard steels (C 0.35–0.10%; Si 0.1–4.5%), and cast irons (C 2% and Si 0.1–7%). The authors confirm that the A3 point rapidly decreases with increase of silicon (disappearing with Si 1.3%), and at the same time rises very slightly on the temperature scale. The temperature at which the A1 transformation takes place rises with the silicon content, this point reaching and then passing the A2 point, whilst becoming gradually less marked. After reaching a given percentage of silicon and for a given rate of variation of temperature, the A1 point completely disappears, and all the carbon of the metal is then found in the state of graphite. The A2 point falls gradually on the temperature scale and becomes gradually less marked as the silicon increases, but it still persists with Si 7%. The authors emphasise the fact that the A2 point can persist when both the A1 and A3 points have disappeared as proving the real and distinctive individuality of the A2 point. As regards the influence of silicon on the solubility of carbon in iron, the results of the tests made with the series of irons with C 2% show that the solubility gradually decreases with rise of silicon, becoming practically nil at 900° C. when the silicon content reaches 5%, and at about 1000° C. with Si 7%. The presence of the silicon reacts, on the one hand, on the temperature limits within which the precipitation of graphite takes place, and, on the other hand, on the rate of transformation of the cementite. Thus, provided that the silicon content exceeds 3%, that the annealing is carried out at above 750° C. but below a certain limit, which is a function of the carbon content, and that the rate of cooling after annealing is very low, the carbon may be wholly precipitated in the graphitic state in forged steels with low or medium

carbon content. Also, the rate of reaction may be influenced to such an extent that in certain irons the graphite precipitation gives rise to a thermal point. This point, which may coexist with the A1 point, possesses special sharpness when the latter has disappeared; it then rises rapidly on the temperature scale, as the silicon content increases. By extrapolating for zero content of silicon from the results obtained, the authors consider that evidence is obtained supporting the double iron-carbon diagram (graphite system and cementite system).—T. St.

*Boron in iron; Determination of*.—J. M. Lindgren. J. Amer. Chem. Soc., 1915, 37, 1137—1139.

Two to three grms. of iron is dissolved in a round-bottom Jena flask of 800 c.c. capacity in 10 c.c. each of nitric acid (sp. gr. 1.4), hydrochloric acid (sp. gr. 1.2), and water. Double the amount of pure calcium carbonate necessary for complete neutralisation is added, all at once, and the mixture vigorously agitated: 250 c.c. to 300 c.c. of CO<sub>2</sub>-free water is added and the mixture boiled under a reflux condenser for half an hour. About 50 c.c. of asbestos fibre, which has been treated with acid and well washed, is added in a wet state, the mixture is boiled, filtered, and the precipitate washed eight or ten times with hot water. The solution is cooled and titrated with N/10 alkali, using phenolphthalein as indicator (compare this J., 1908, 1179).—H. R. D.

*Grey cast iron and steel; Relative corrodibilities of*.—J. N. Friend and C. W. Marshall. Iron and Steel Inst., May, 1915. [Advance proof.] 5 pages.

Bars of a typical grey cast iron and of a mild open-hearth steel were subjected to tap-water, salt-water, alternate wet and dry and hot and cold, and sulphuric acid tests (see also this J., 1912, 493). In the alternate wet and dry tests the cast iron was usually decidedly superior to the steel; complete immersion tests gave almost identical results for the two metals, except with salt water, which attacked steel more strongly. In acid solutions cast iron was corroded to a much greater degree than steel.—W. R. S.

*Iron; Corrosion of—in aqueous solutions of inorganic salts*.—J. N. Friend and P. C. Barnett. Iron and Steel Inst., May, 1915. [Advance proof.] 17 pages.

The loss of weight of pure electrolytic iron foil was determined after immersion in solutions of aluminium, ammonium, ferrous, magnesium, manganese, potassium, and sodium sulphates, barium, magnesium, potassium, and sodium chlorides, and potassium and sodium nitrates, the results being tabulated for each salt at various temperatures and concentrations. Concentrated solutions of most of the salts were less corrosive than distilled water. As a rule, the rate of corrosion rose with the temperature, whilst the corrosion factor, i.e., corrosion in the salt solution relatively to that in distilled water, fell. In general, and conversely, the more concentrated the solution of any salt, the lower its inversion temperature, i.e., the temperature at which the solution has the same corrosive action as distilled water. The critical concentration corresponding to maximum corrosion tends to fall with rise of temperature.—W. R. S.

*Rust; Removal of—by means of chemical reagents*.—J. N. Friend and C. W. Marshall. Iron and Steel Inst., May, 1915. [Advance proof.] 7 pages.

The object of the experiments was to remove all the rust without dissolving any iron. Sheets of mild open-hearth steel were moistened, exposed

to the air for one month, and thoroughly cleaned with emery paper, the mean loss being taken as a standard of comparison with the results obtained by solvents. Sodium citrate solution acted very slowly and dissolved much metal. Boric acid solution had a good cleaning action, but the loss in weight was 75% more than the standard. Aluminium sulphate solution (10%) gave an excellent metallic surface, but none of the reagents left the metal unattacked.—W. R. S.

*Steel ingots; Supplementary notes on the forms in which sulphides may exist in*.—J. O. Arnold and G. R. Bolsover. Iron and Steel Inst., May, 1915. [Advance proof.] 4 pages. (See also this J., 1914, 551).

THE addition of about 0.05% of aluminium to molten steel containing about 0.5% S prevented segregation, and gave rise to areas showing the "eutectic" structure of decomposed solid solution or compound ( $x\text{Fe}_3\text{MnS}$ ). These areas were surrounded by regions of ferrite (and pearlite) relatively free from sulphides. A slowly cooled steel to which no aluminium had been added showed marked segregation of carbon, sulphur, and manganese. Micrographically the manganese sulphide was observed to be segregated into relatively large rounded masses, quite different from the eutectic structure formed when aluminium had been added.—T. St.

*Special steels; The thermo-electric properties of*.—E. L. Dupuy and A. M. Portevin. Iron and Steel Inst., May, 1915. [Advance proof.] 30 pages.

THE investigation was made with 27 nickel steels (Ni 2—30, C 0.1—1.0%), 17 chromium steels (Cr 1—32, C 0.06—1.0%, Si up to 0.75%), 4 manganese steels (Mn 0.4—12, C 0.9%), 7 aluminium steels (Al 2—15, C 0.1—0.9%), 7 silicon steels (Si 0.4—5.5, C 0.2—0.9%), 10 tungsten steels (W 0.4—20.7, C 0.1—0.9%), 8 molybdenum steels (Mo 0.5—4.5, C 0.2—0.8%), 7 very pure ordinary carbon steels (C 0.07—1.6%), and electrolytic iron. Thermo-electric measurements were made between  $-78^\circ$  and  $0^\circ\text{C}$ . and between  $0^\circ$  and  $100^\circ\text{C}$ ., the test-pieces being annealed by heating to  $900^\circ$ — $1000^\circ\text{C}$ . for 3 hours and cooling during 5—9 hours; or hardened by heating to  $950^\circ$ — $1000^\circ\text{C}$ . during 11—13 mins. and quenching in water at  $11^\circ$ — $18^\circ\text{C}$ . In addition to being discussed as separate series, the results are considered collectively in the light of the investigations of Flakén (Ann. Physik, Series 4, 1910, 32, 291) who found that, in the case of a solid solution, a portion of the curve representing thermo-electric force in terms of composition resembles an arc of hyperbola having its concave side either upwards or downwards; whereas the curve for a mixture of two phases is practically a straight line. The thermo-electric force being positive or negative, the definite compounds constituting solid solutions may be either represented in the curves by maxima or minima, or situated along a branch in the form of an S and marked only by a point of inflexion. On this basis, the special steels examined were found to fall into two groups, namely, (1) those containing nickel, manganese, silicon, and aluminium, which gave continuous curves resembling a U or part of a U in shape, indicating the added metals to be in a state of solid solution in the steels; and (2) those containing chromium, tungsten, and molybdenum, the curves for which—although indicating (by the sudden initial fall) the passage of the added metal into solid solution—on the whole resemble an S in form: "double carbide" steels occur in the latter group. It is considered possible to form an idea as to the limit of solubility, in the solid state, of the special metals added to steels, the carbide

becoming isolated (when saturation is reached) either as a constituent of the pearlite or as a super-eutectic constituent.—W. E. F. P.

*Vanadium in steel; Simplified ferrous sulphate method for the determination of—* G. T. Dougherty. J. Ind. Eng. Chem., 1915, 7, 419—420.

From 2 to 4 grms. of drillings is heated nearly to boiling with 80 c.c. of water and 10 c.c. of concentrated sulphuric acid, 40 c.c. of nitric acid of sp. gr. 1.2 is added, and the mixture is boiled till all nitrous fumes are expelled. After cooling, the solution is treated with 60 c.c. of sulphuric acid (1:2), diluted to 450 c.c., and, after addition of 3 c.c. of a freshly prepared 1% solution of potassium ferricyanide, titrated rapidly with N/20 ferrous ammonium sulphate, with constant stirring, until a dark blue colour first appears; 0.4 c.c. is deducted from the volume of ferrous solution used in the case of steels containing up to 0.5% C, 0.5 c.c. for 0.6—0.7% C, and 0.6—0.9 c.c. for 0.9—1.25% C. When the steel contains more than 0.5% C, it is advantageous to add 60 c.c. of sulphuric acid (1:2) and 5—8 grms. of ammonium persulphate after boiling with nitric acid, and continue to boil for 15 mins., before diluting and titrating; the correction is then only 0.35 c.c. for steels with less than 0.5% C, but the same as before for higher carbon contents. The ferrous solution may be standardised against N/10 permanganate; the iron value of the permanganate multiplied by 0.917 gives the vanadium value. The method is accurate to within 0.01% V.—A. S.

*Ferrovanadium; Technical analysis of—* A. Heinzelmann. Chem.-Zeit., 1915, 39, 285—287.

THE finely-divided sample (0.5 gm.) is treated with concentrated hydrofluoric acid (10 c.c.), nitric acid is added after the evolution of hydrogen has ceased, and the mixture is heated on a water-bath and then evaporated to dryness. The residue is heated with sulphuric acid to expel all the nitric acid, the sulphuric acid solution diluted with water, boiled, and made up to 200 c.c. Fifty c.c. of this solution is treated with hydrogen peroxide, diluted to 250 c.c., neutralised, and boiled after the addition of 50 c.c. of concentrated sulphurous acid solution. To the boiling solution is now added 20 c.c. of 30% sulphuric acid, the boiling is continued for about 20 minutes while a current of carbon dioxide is passed through the solution, and the iron and vanadium together are then titrated with N/10 permanganate solution. A slight excess of N/10 ferrous ammonium sulphate solution is added to the titrated solution (spot-tests with potassium ferricyanide are used as indicator), the excess is titrated with N/10 permanganate solution, and the titration with the latter solution then continued until a slight reddish-brown coloration is obtained. The number of c.c. of N/10 permanganate solution used in the last titration multiplied by 0.00512 gives the quantity of vanadium present, and the iron is found by difference.—W. P. S.

*Iron oxides; Reduction of—by platinum.* R. B. Sosman and J. C. Hostetter. J. Wash. Acad. Sci., 1915, 5, 293—303.

To demonstrate the absorption of iron from magnetite by platinum under very low oxygen pressure, about 1 gm. of natural magnetite from Mineville, N.Y., was heated to 500° C. in a new platinum crucible in a platinum-rhodium tube vacuum furnace and cooled. The gas produced was pumped out, and the charge then heated to between 600° and 1200° C. The changes in weight of charge and crucible indicate that oxygen is liberated and metallic iron is dissolved by the platinum. At 10.2

mm. oxygen pressure at 1200° C., under which conditions magnetite is nearly completely oxidised to ferric oxide, the crucible gained 1.6 mgrms. Platinum acts on both hæmatite and magnetite at 1200° C. under low pressure of oxygen, absorbing iron and causing evolution of oxygen. Platinum crucibles in which ferric oxide is ignited in air in ordinary analysis, do not take up such amounts of iron as are mentioned. The phenomena observed explain the very common occurrence of small amounts of iron in platinum.—H. R. D.

*Litharge; Precious metals in—* J. Loey. Chem.-Zeit., 1915, 39, 287. (See also Michel, this J., 1915, 285.)

FROM 1200 grms. of litharge the author obtained a weighable quantity of gold. Platinum could not be detected in the litharge. The assumption that colloidal platinum occurring in ores cannot be determined by the ordinary assay methods, is disproved by the results of experiments in which colloidal gold and platinum were added to ores and the latter then analysed; from 98 to 99.6% of the two metals was recovered.—W. P. S.

*Gold-bearing cyanide solutions; Morro Velho method of assay of—* D. M. Levy and H. Jones. Inst. Min. and Met., May 20, 1915. [Advance proof.] 3 pages.

THE method has been in regular use at a plant at Morro Velho, Brazil, for the daily control of solutions passing through the zinc boxes and other solutions. It is based on the precipitation of gold by means of zinc shavings from the boiling solution in presence of suitable quantities of silver and lead salts, to yield with the gold a parting alloy and produce a zinc-lead couple which facilitates the complete separation of metallic gold from solution. The process is as follows: To 453.6 c.c. of rich solution (before passing the zinc boxes) are added in succession 15 c.c. of silver solution, 10 c.c. of sodium cyanide solution, and 5 c.c. of sodium plumbite solution, and the mixture is shaken in a flask of 1½ litres capacity. 20 grms. of zinc shavings is then placed in the solution and the whole gently boiled for 30 minutes and then filtered. To the residual zinc in the flask a little water is added, and 70 c.c. of commercial hydrochloric acid to dissolve the zinc. The contents of the flask are filtered, and both filter and flask are well washed with water. The wet filter is transferred to a No. 10 assay crucible, dried slowly, and carbonized at a moderate heat. The residue is mixed with a fluxing charge and assayed in the usual manner. The following stock solutions are used:—Silver solution: 14.4 grms. of silver nitrate dissolved in water, sodium cyanide added slowly till the precipitate first formed is just redissolved, and the solution then diluted to one litre. Sodium cyanide solution: 100 grms. of sodium cyanide dissolved in water and diluted to one litre. Sodium plumbite solution: 26 grms. of lead acetate dissolved in water, sodium hydroxide solution added until the precipitate first formed just redissolves, and the solution diluted to one litre.—H. R. D.

*Gold; Concentration of—in bottoms in the converter.* H. F. Collins. Inst. Min. and Met., May 20, 1915. [Advance proof.] 6 pages.

SUCCESSFUL attempts have been made to utilise the gold contents of converter copper not rich enough in precious metals to make electrolytic refining profitable, by means of a partial oxidation of white metal to "pimple" metal and "bottoms," and the separation of the latter before completing the oxidation. The success of the operation depends primarily upon running the charge hot, not losing time in skimming, and not adding too much cold scrap after skimming, so that the reduced copper is at a temperature much above

the melting point of the "pimple" metal; otherwise it is liable to chill in the tap-hole. The converter is provided with a cast iron tapping block fixed opposite the tuyères, having a tap hole  $1\frac{1}{4}$  in. in diameter. When lining is being rammed, a  $\frac{1}{2}$  in. steel bar is pushed through the hole up to within an inch of the mould, so that the lining may be rammed round it as tightly as possible, and when moulds are removed the bar is withdrawn at the same time to facilitate "drying out." In practice it is easy to recover in the form of bottoms, 70% of the gold originally existing in the charge, the amount of bottoms being 9% of the total weight of converter copper produced.

—H. R. D.

*Copper leaching; Discussion on*—. Amer. Electrochem. Soc., April, 1915. Met. and Chem. Eng., 1915, 13, 319–324. (See also this J., 1913, 677; 1915, 555.)

U. WEDGE stated that sulphide ores from Ajo, Arizona, ground to 20- or 30-mesh, could be treated successfully by a sulphatising roast not much above red heat. By roasting a mixture of oxide and sulphide ore, or iron pyrites, the necessity for constructing an acid plant could be obviated. G. W. Van Arsdale said that at Douglas, Arizona, sulphatising roasting was now used in conjunction with electrolysis, iron being found not to interfere with the deposition of the copper if kept in the ferrous state by means of sulphur dioxide. Graphite anodes were used. R. F. Bacon stated that the installation and running cost of an electrolytic plant were rather high, and the whole of the copper in solutions obtained by leaching oxidised copper ores was not deposited by the current; a careful adjustment was therefore required, so as to secure a cycle of operations. F. J. Pope had found that most of the iron (90%) and alumina (65–75%) might be precipitated from the electrolyte, together with 50% of the manganese and all of the arsenic, antimony, and bismuth which might be present, by agitating with air at 90° C., and gradually adding finely ground copper oxide in the form of high-grade carbonate ore or roasted concentrate or matte. The precipitation was complete after 3½ hours, when the liquor was filtered and electrolysed. Antimonial lead anodes were used.—W. R. S.

*Cobalt; Electroplating with*—. H. T. Kalmus, C. H. Harper, and W. L. Savell. J. Ind. Eng. Chem., 1915, 7, 379–399.

This is an abstract of a report to the Canadian Department of Mines, containing an account of a large number of experiments with different cobalt plating solutions both in the laboratory and in the plating department of the Russell Motor Car Co., Toronto. Of the solutions tried the best results were obtained with the following:—1b. 200 grms.  $\text{CoSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  per litre; sp. gr. 1.053 at 15° C. 111b. 312 grms.  $\text{CoSO}_4$  and 19.6 grms.  $\text{NaCl}$  to 1000 c.c.  $\text{H}_2\text{O}$ , the solution being nearly saturated with boric acid; sp. gr. 1.25 at 15° C. These solutions have a considerably higher electrical conductivity than standard commercial nickel plating solutions, and hence may be operated at a lower voltage for a given speed of plating. When used for very rapid plating a higher voltage is required than is used for most nickel plating baths, but for a given amount of work the power consumption is less than for nickel. Firm, adherent, uniform deposits of cobalt can be obtained on brass, iron, steel, copper, tin, German silver, lead, and Britannia metal with solution 1b at a speed at least four times, and with solution 111b at least fifteen times greater than that possible with nickel solutions. The cobalt coating is much harder than a nickel coating, and for many purposes the same protective effect as is obtained

with a given quantity of nickel can be obtained with one-quarter of that quantity of cobalt. The cobalt solutions give less trouble in use than nickel solutions, and are also superior in "throwing power," i.e., capacity to coat deeply indented or grooved parts. With solution 111b, ornamental work on brass, copper, tin, or German silver can be coated in 1 min. so as to withstand the usual commercial tests, including buffing, and satisfactory deposits can be obtained in 15 mins., even for objects exposed to severe atmospheric influences. Thick deposits of cobalt, e.g., on electro-types and electro-dies, are much superior to those of nickel. The composition of the baths remains constant during use, and less ageing is required than with commercial nickel solutions. Skates plated with cobalt in solution 111b for 3 mins. at 90–100 amp. per sq. ft. of cathode surface, have proved superior to nickel-plated skates in actual use. Electrodeposited cobalt can be readily buffed to a brilliant white, lustrous surface with a slightly bluish cast. In the report of the commercial tests it is stated that the cost of metal for plating a given quantity of work would be considerably less with cobalt than with nickel, whilst on account of the higher speed of plating it would be possible to extend the use of automatic apparatus for conveying the material through the bath to a much greater variety of articles than with nickel, and reduce the labour cost 75%.—A. S.

*Lead deposits; Electrolytic*—. F. C. Mathers and A. McKinney. Amer. Electrochem. Soc., April, 1915. Met. and Chem. Eng., 1915, 13, 328. (See also this J., 1914, 358, 1158.)

A SMOOTH, coherent deposit may be obtained from lead nitrate with a bath of the following composition: lead nitrate, 10%; acetic acid, 2.5–5%; residue from extraction of aloin from Curaçao aloes, 1%; the latter is dissolved in the acetic acid and added to the lead solution without filtering. A current of 3.7 amp. per sq. ft. may be used. Nitrate baths do not promise to be of practical use, as they deteriorate after 6–7 weeks.

—W. R. S.

*Magnesium; Electrolytic preparation of*—. F. C. Frary and H. C. Berman. Amer. Electrochem. Soc., April, 1915. Met. and Chem. Eng., 1915, 13, 324–325.

In the electrolysis of molten magnesium potassium chloride in a graphite crucible, dark spots are found in the electrolyte on cooling. They are stated to consist of magnesium suboxide, and their formation to be due to the presence of magnesium oxide. The compound could not be obtained by the action of magnesium upon the oxide, being apparently only formed under the conditions of electrolytic reduction. The electrolyte should be free from magnesium oxide.

—W. R. S.

*Sulphates; Formation and decomposition of*—during roasting. B. Dudley, jun. Met. and Chem. Eng., 1915, 13, 303–308. (See also this J., 1915, 496.)

THE rate at which a sulphate is formed or decomposed depends upon the rate of diffusion of sulphur trioxide through the contact film surrounding the particle of oxide or sulphate, and on the difference between the dissociation tension of the sulphate and the partial pressure of sulphur trioxide in the gaseous phase. The rate of decomposition, in a current of dry air, of a sulphate (e.g.,  $\text{NiSO}_4$ ) dissociating directly to oxide was found to be nearly constant; under the same conditions, the rate of decomposition of a sulphate (e.g.,  $\text{CdSO}_4$ ) dissociating first to a basic compound showed a marked decline at the point at which the conversion into the basic sulphate was complete. The rate

of dissociation of a sulphate further increases with that of sulphur trioxide:  $2\text{SO}_3 = 2\text{SO}_2 + \text{O}_2$ ; this is mainly determined by the presence or absence of catalysts, such as ferric oxide. This was proved by heating dehydrated aluminium sulphate in a tube furnace, with and without iron oxide, and determining the loss of weight at regular intervals: the rate of decomposition of the sulphate was in all cases accelerated, and the ratio of sulphur trioxide to dioxide in the evolved gas lowered, by the presence of ferric oxide.—W. R. S.

*Metals; The elastic strength of*—. F. C. Thompson. Faraday Soc., May 11, 1915. [Advance proof.] 13 pages.

THE theory is advanced that the elastic strength of metals is due to surface tension effects of the amorphous "cement" between the crystals, and not to the strength of the cement itself, as hitherto supposed. On this hypothesis, the conditions governing elastic limit are essentially different from those governing maximum stress; the attraction between the crystals being inversely proportional to the distance between them, the elastic strength of the metal is lowered as the thickness of the intercrystalline cement is increased, and the superiority of fine- over coarse-grained metal is due to the increased area across which the attraction occurs. The theory is considered quantitatively and qualitatively, evidence in support of it being deduced from the mechanical properties of pure copper, zinc, antimony, silver, lead, tin, and iron under different conditions, and from variations in the electrical conductivity of iron after different heat treatments.—W. E. F. P.

#### PATENTS.

*Blast furnace; Mode of charging a*—, and *construction of the furnace for that purpose*. R. Wright, Jarrow-on-Tyne, and J. Calderwood, South Bank. Eng. Pat. 9939, July 4, 1914.

IN a blast-furnace having a central ore shaft, around the middle portion of which smaller shafts are disposed for the admission of fuel, each of the latter is provided with a metal charging-tube and an inlet for cold air, and discharges into the combustion zone of the furnace at a point directly above a blast tuyère, so that the gases ascending the ore shaft are cooled sufficiently by contact with the undecomposed and relatively cool fuel, to obviate premature fusion of the charge.

—W. E. F. P.

*Iron or steel; [Noncorrodible] alloys of*—. B. Talbot, Middlesbrough. Eng. Pats. 10,582, April 29, and 10,877, May 2, 1914.

THE alloys are prepared by adding to molten iron or steel 0.3—3.0% of copper, casting the metal, rolling the ingots into sheets, piling the latter, and forming the heated pile into the desired shape by hammering, cogging, or rolling. A similar process may be used for the manufacture of articles containing a higher percentage of copper at the exterior than in the interior.—W. E. F. P.

*Mild steel; Production of an oxide coating on utensils and the like made of*—. C. K. Haefner. Ger. Pat. 282,114, April 10, 1913. Addition to Ger. Pat. 257,290.

OXIDES of nickel, copper, and cobalt, in conjunction with manganese dioxide, are used in place of, or together with, the iron oxide specified in the chief patent (this J., 1913, 492).—A. S.

*Metal from metal-bearing material; Process for the extraction of*—. W. H. James, Johannesburg, Transvaal. Eng. Pat. 9848, April 21, 1914.

A SOLUTION of a salt, e.g., sodium chloride, is electrolysed, portions of the electrolyte being withdrawn separately in the vicinities of the electrodes.

The material is leached with the liquor from one of the electrodes, usually that from the anode, and the metal-bearing solution is withdrawn and precipitated with liquor from the other electrode.

—W. R. S.

*Ores; [Electrolytic] process and apparatus for treating*—. L. E. Porter, Los Angeles, Cal., Assignor to H. E. Stock, Casper, Wyo. U.S. Pat. 1,136,483, April 20, 1915. Date of appl., Aug. 27, 1914.

A COMBINED electrolytic cell and agitator is subdivided by porous diaphragms into anode and cathode compartments; a pulsating current of electrolyte containing suspended ore is injected at the bottom of the anode compartment, from which it flows intermittently into the cathode compartment, in which the values extracted in the former are deposited.—W. R. S.

*Blast-furnace construction*. T. E. Thomas, Niles, Ohio. U.S. Pat. 1,137,244, April 27, 1915. Date of appl., July 17, 1913. Renewed Sept. 21, 1914.

A PORTION of the stack above the bosh of the furnace consists of an inner and an outer wall between which a water-jacket is interposed, consisting of a single metal shell or a series of box-like members of segmental form.—W. E. F. P.

*Ore concentration*. L. A. Wood, and Minerals Separation Ltd., London. Eng. Pat. 10,312, April 25, 1914.

POWDERED sulphide ores suspended in water, preferably acidified, but free from oil or frothing agent, are subjected to agitation in the lower zone of the ore pulp, and air is admitted below the agitator. The upper zone of the pulp is maintained in a state of comparative quiescence, whereby the finely-divided bubbles of air attach themselves preferentially to selected particles of the ore, such as sulphides, and rise towards the surface of the liquid, discharging their mineral load into a trough or receiver below the surface.—B. N.

*Ores and more particularly slimes; Water concentration of*—. W. M. Martin, Redruth. Eng. Pat. 15,442, June 27, 1914.

THE ore, mixed with water, is made to flow over a stationary or continuously moving acting surface, consisting of a material which only slightly hinders the movement of the larger particles, but is provided with regular or frosted flutings adapted to cause greater hindrance to the smaller particles. The apparatus may be in the form of a wheel, divided into compartments, one surface of each compartment consisting of fluted or frosted glass.

—B. N.

*Furnaces; Gas heated annealing*—. S. N. and E. R. Brayshaw, Manchester. Eng. Pat. 10,630, April 30, 1914.

SEVERAL burner openings are arranged in the bottom of the furnace, each surmounted by a hearth slab upon which the flame is directed. A longitudinal channel is provided at each side of the furnace, below the hearth slabs, for collecting scale or slag, thus preventing impediment to the passage of the flame. A swivelling burner is pivoted below each opening, which is closed by a sliding damper held in position by the burner when the latter is not in operation.—B. N.

*Furnace; Reverberatory— for heating, annealing, and like purposes*. C. Glossop, Sheffield. Eng. Pat. 10,111, April 24, 1914.

TO ensure uniform heating, the furnace is provided with a perforated bed upon which the charge is placed close to the fire-bridge, and centrally within

the heating chamber, so that the heating gases are caused to traverse the space around the charge before escaping by a vertical down-flue below the bridge.—W. E. F. P.

*Furnaces; Crucible and like* — A. C. Ionides, jun., London. Eng. Pat. 15,778, July 1, 1914.

THE combustion chamber is surrounded by a spiral flue, through which the products of combustion pass to the atmosphere. The air or gas for combustion, or both, are preheated on their way to the combustion chamber by passing through spiral conduits.—B. N.

\* *Furnace; Metallurgical* — U. Wedge, Ardmore, Pa. U.S. Pat. 1,137,559, April 27, 1915. Date of appl., Oct. 22, 1913.

IN a furnace of the superposed chamber type, one of the circular hearths, consisting of radial sections, has alternate segments recessed on the under side, and the recesses filled with electrically resistant material. An electric current is passed through each filling, from which heat is radiated to the charge on the hearth below.—W. E. F. P.

[*Copper sulphide ores; Method of treating* — R. Baggeley, Pittsburgh, Pa. U.S. Pat. 1,135,488, April 13, 1915. Date of appl., June 17, 1913.

IN smelting sulphide ores containing copper, gold, and silver, the flue gases are passed over fresh ore so as to preheat and enrich it, and are then led in small streams into a deep bath containing a neutralising agent, such as milk of lime.—W. R. S.

*Bearings or wearing surfaces.* The British Thomson-Houston Co., Ltd., London. From General Electric Co., Schenectady, N.Y., U.S.A. Eng. Pat. 10,872, May 2, 1914.

CAST-IRON bearings, etc., are provided with "sherardised" wearing surfaces.—W. E. F. P.

*Zinc-producing furnace.* A. Zavelberg, Hohenlohehütte, Germany. U.S. Pat. 1,136,304, April 20, 1915. Date of appl., Aug. 25, 1914.

THE furnace comprises two or more superposed rows of shafts containing retort chambers, and a preheater above the upper shaft. The preheater is connected with the upper shaft, and this with the lower one, by means of flues. Packing layers of finely pulverised silica are provided between the walls of adjoining retort chambers.—W. R. S.

*Lead and zinc; Separation of the sulphur compounds of* — from other ores. E. Langguth. Ger. Pat. 282,131, Oct. 8, 1913.

THE mixed ores, after addition of oil or other suitable organic compound, are treated with a solution of zinc chloride acidified with hydrochloric acid. Sulphides of zinc and lead sink in the solution, whilst other ores float.—A. S.

*Muffle for the distillation of zinc.* M. Engels, Berlin. U.S. Pat. 1,138,519, Apr. 20, 1915. Date of appl., Feb. 11, 1915.

THE muffle is composed of a fireclay base containing 1–10% of zirconia.—W. E. F. P.

*Zinc; Process and electric furnace for obtaining volatile metals, especially* — M. Breslauer. Ger. Pat. 282,141, Jan. 30, 1913.

THE charge is introduced into a carbon tube, preferably vertical, which serves as a resistance heater, and the zinc vapour and carbon monoxide pass through slots or perforations in the tube, into the space between it and the walls of the furnace. The zinc condenses in this space in the liquid state, and the carbon monoxide escapes through openings in the walls.—A. S.

[*Gold and silver ores; Process of treating* —

E. M. Hamilton, Bristol, Assignor to C. Butters, Oakland, Cal. U.S. Pat. 1,136,872, Apr. 20, 1915. Date of appl., June 5, 1914.

A MIXTURE of slimes and cyanide solution is made alkaline with lime, the latter precipitated so as to produce caustic alkali in the solution, the mixture filtered, and the precious metals precipitated from the solution by means of aluminium.—W. E. F. P.

*Zinc; Process for rendering spongy, electro-deposited* — stable. Chem. Fabr. Griesheim-Elektron. Ger. Pat. 282,234, June 29, 1913.

SPONGY zinc is freed from adhering electrolyte and then dried, with exclusion of air.—A. S.

*Aluminium and heavy metals; Production of non-corrodible objects of* — A. Lang. Ger. Pat. 282,328, Dec. 4, 1913.

THE parts made of aluminium and of the other metal respectively, before and after they are brought together, are treated by any suitable method to produce on them coatings of oxide or sulphide of the same or of another metal, which are not attacked by dilute acids or alkalis and are capable of withstanding heat and mechanical treatment.—A. S.

*Iron oxide ores and iron oxides; Process of reducing* — H. C. Alford, Mobile, Ala., U.S.A. Eng. Pat. 11,224, May 6, 1914.

SEE U.S. Pat. 1,097,156 of 1914; this J., 1914, 698.

*Steel; Process of refining* — and producing high-phosphorus slag. A. Vögler, Assignor to Deutsch-Luxemburgische Bergwerks- und Hütten-A.-G., Dortmund, Germany. U.S. Pat. 1,137,681, Apr. 27, 1915. Date of appl., Apr. 10, 1912.

SEE Eng. Pat. 19,640 of 1911; this J., 1912, 496.

*Ores; Treatment of subdivided* — for agglomerating or reducing them, and apparatus therefor. G. Gröndal, Djursholm, and H. Nilsson, Grangårde, Sweden. Eng. Pat. 20,361, Sept. 30, 1914. Under Int. Conv., Oct. 11, 1913.

SEE Fr. Pat. 473,218 of 1914; this J., 1915, 558.

*Crucible and like furnace.* A. C. Ionides, jun., London. U.S. Pat. 1,138,482, May 4, 1915. Date of appl., Oct. 4, 1912.

SEE Eng. Pat. 2839 of 1912; this J., 1913, 198.

*Process for effecting chemical reactions at high temperatures.* [Melting metals.] Ger. Pat. 281,004. See I.

## XI.—ELECTRO-CHEMISTRY.

*Furnace; Vacuum [electric] — for the measurement of small dissociation pressures.* R. B. Sosman and J. C. Hostetter. J. Wash. Acad. Sci., 1915, 5, 277–285.

THE furnace comprises a tube acting as the heating element, within which is maintained a high vacuum, and a large, outer, water-cooled iron jacket, within which is maintained an "ordinary" vacuum, surrounding the furnace tube. An alternating current at about 2 volts is supplied to the furnace tube by water-cooled leads, passing through a stone base forming the bottom of the outer jacket. The furnace tube, consisting of an alloy of 80% Pt and 20% Rh, is surrounded by a magnesia tube, and the lower end is sealed into a water-cooled steel tube, passing through the stone

base, whilst the upper end is extended by a steel tube sealed into a glass tube. The charge is carried within the furnace tube by a small platinum crucible suspended by platinum wires from the top of the glass tube, from which is also suspended a Pt-Rh thermo-element. The glass tube is connected on one side to suitable pumps and an oxygen supply, and on the other to three pressure gauges, by means of which pressures ranging from 0.000001 mm. Hg to 2.5 atmos. may be measured. Experiments made on the oxygen pressures produced by 0.5 gm. charges of ferric oxide, heated at different levels in the furnace tube, have shown that there is a range of 25–30 mm. in the tube within which the temperature is uniform within 1° at about 1500° C. The heating current is supplied from the secondary of a transformer, a motor-generator supplying the primary, and the voltage of the generator is regulated by its field current. With a reasonably constant voltage applied to the motor, the temperature is practically constant, about 580 amps. at about 1.8 volts being required for a temperature of 1450° C.—B. N.

*Lead; The anodic solution of —.* N. M. Bell. Faraday Soc., May 11, 1915. [Advance proof.] 12 pages.

THE electrolysis of lead acetate, sodium acetate, potassium hydrogen tartrate, hydrofluosilicic acid, potassium bromide, and sodium thiosulphate solutions, at various current densities, has shown that an anode of pure lead loses more than may be expected, according to Faraday's law, for divalent ions. With lead acetate, potassium hydrogen tartrate, hydrofluosilicic acid, and potassium bromide, by the use of a blank electrode and by means of time experiments, this extra loss is shown to be due to chemical solution and mechanical detachment in cleaning, and not to the lead dissolving as univalent ions. In the case of sodium acetate and thiosulphate solutions, there is evidence of univalent lead ions in aqueous solution, to the extent of 2.8% in the former and 5.8% in the latter.—B. N.

*Cadmium electrode; Reproducibility of the —.* F. H. Getman and V. L. Gibbons. J. Amer. Chem. Soc., 1915, 37, 953–970.

THE measurements of the E.M.F. in concentration cells of methyl alcoholic solutions of cadmium iodide show that neither fresh cast rods of cadmium nor spongy cadmium deposited electrolytically are reproducible. Photomicrographs indicate a change in crystalline form. Rods of cadmium which have become grey by standing in cadmium iodide solution, and crystalline deposits of cadmium on cadmium rods or platinum give a constant E.M.F. when measured against an unpolarisable electrode; the E.M.F. of the crystalline is slightly higher than that of the grey cadmium.—H. R. D.

*Electrochemical oxidation of hydrazine sulphate and ammonium hydroxide.* Turrentine and Olin. See VII.

#### PATENTS.

*Bleaching liquor; Electrolytic apparatus for the production of —, and for other purposes.* A. Holliday, Wimbledon, and G. E. Ward, Stratford. Eng. Pat. 1672, Feb. 2, 1915.

THE electrodes are provided with elongated insulated lugs, which are secured to the top plate of a frame by metal studs, a rubber washer being interposed between the upper ends of the lugs and the plate. The bus bars and terminal fittings are enclosed in a protective cover. When the frame is lifted out of the vat any electrode may be removed separately from below, without removing

the top plate of the frame or disconnecting the other electrodes. A deflecting plate is provided to ensure efficient circulation of the liquid.—B. N.

*Electrolyte for primary batteries.* A. P. Manchester, Providence, R.I. U.S. Pat. 1,187,226, April 27, 1915. Date of appl., Dec. 26, 1912.

ONE pound of an alkali hydroxide, such as sodium hydroxide, of about 28° B. (sp. gr. 1.24), is combined with 273.1 grs. of vegetable starch at about 180° F. (82° C.)—B. N.

*Electrodes of manganese dioxide; Manufacture of —.* Siemens und Halske A.-G. Ger. Pat. 282,225, Feb. 22, 1914. Addition to Ger. Pat. 221,130.

PYROLUSITE is finely ground, freed from impurities by treatment with dilute sulphuric acid or other acid which does not decompose manganese dioxide, then well washed, dried, moistened with manganese nitrate solution, and formed into electrodes as described in the chief patent (see U.S. Pat. 935,109; this J., 1909, 1144).—A. S.

*Preparation of hydrocyanic acid by the circulating passage of a gas mixture through an expanded high-potential arc.* Eng. Pat. 11,107, See VII.

[Electrical] sterilisation of liquids. Eng. Pat. 9567, See XIXb.

#### XII.—FATS; OILS; WAXES.

*Calcium and magnesium soaps; Formation and solubility of —.* J. Zink and R. Liere. Z. angew. Chem., 1915, 28, 229–232. (See also Haupt, this J., 1915, 39.)

THE reaction between soluble calcium or magnesium salts and the alkali salts of fatty acids proceeds quite normally with the formation of an alkali salt and an insoluble soap, the reaction being practically quantitative whichever calcium or magnesium salt is employed. One litre of distilled water at ordinary temperature dissolves about 41, 28, and 91 mgrms. of calcium stearate, palmitate, and oleate and 78, 62, and 103 mgrms. of magnesium stearate, palmitate, and oleate, respectively; the solubility in each case is considerably increased by the addition of small amounts (0.5 and 1 gram. per litre) of sodium chloride.—T. C.

*Determination of fat [in animal substances, etc.].* Rosenthal and Trowbridge. See XIXa.

#### PATENTS.

*Linseed oil; Preparation of a substitute for boiled —.* E. F. Waentig. Ger. Pat. 281,452, Feb. 28, 1913. Addition to Ger. Pat. 272,465. (See also Ger. Pats. 273,347 and 276,430; this J., 1914, 703, 1020.)

AN animal oil is heated to 280° C., then the supply of heat is cut off, and the containing vessel is insulated to minimise loss of heat. The temperature rises at first, and when subsequently it falls again to 260°–285° C., the material is treated with superheated steam as described in the chief patent (this J., 1914, 604).—A. S.

*Soaps; Process for the manufacture of hard —.* J. Leimdörfer, Budapest. U.S. Pat. 1,138,230, May 4, 1915. Date of appl., March 28, 1912.

SEE Fr. Pat. 442,248 of 1912; this J., 1912, 936.

*Food product [from cottonseed oil].* U.S. Pat. 1,135,351. See XIXa.

**XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.**

*Pigments; Rapid test for fineness of paint*——  
C. D. Holley and J. C. Brier. Oil, Paint, and Drug Rep., May 10, 1915.

THE Multi-Metal Separating Screen Co. of New York has succeeded in making 350-mesh screens (122,500 apertures per sq. in.) of vanadium-bronze wire about 0.001 in. diam., with which different operators, using different screens, can obtain concordant results. These screens have proved of value for testing the fineness of pigments. The particles passing through can be ground economically in a paint mill, but those retained on the screen, even when present in relatively small amounts, rapidly reduce the efficiency of the mill. The sample (25 grms.) is washed through the screen by means of water, using a soft brush to break up lumps. Calcium and manganese oxides, as used by varnish manufacturers, can be tested in a similar manner, using naphtha instead of water. The results obtained with consignments of various commercial pigments varied greatly in many cases from those yielded by the small sample used as a basis of purchase.—A. S.

*Rosin; Use of types of tinted glass for grading*——  
Oil, Paint, and Drug. Rep., May 10, 1915.

A SET of  $\frac{1}{8}$  in. tinted glass cubes prepared by the U.S. Bureau of Chemistry has been adopted by the Savannah Board of Trade as standards for the various grades of rosin, with the exception of grades D and B. The component colours of the glass types on the Lovibond tintometer scale together with the corresponding grades of rosin are:—

Grade.	Colours.		Grade.	Colours.	
	Yellow.	Red.		Yellow.	Red.
WW ..	20	2-1	I ....	40	7-6
WG ..	20	2-5	H ....	45	9-4
N ....	25	3-3	G ....	50	15-5
M ....	30	4-5	F ....	75	34-5
K ....	35	5-8	E ....	100	52-5

The use of the new standards is to come into operation on June 14, 1915.—A. S.

**PATENTS.**

*Phenolic condensation products.* H. Wade, London. From S. Karpen and Bros., Chicago, U.S.A. Eng. Pat. 9292, April 14, 1914.

A REACTION under practically anhydrous conditions is effected between an anhydrous methylene body, such as hexamethylenetetramine, or hydrobenzamide, a phenol, and a phenolic compound with alkylated hydroxyl groups, such as anisol, phenylmethyl ketone, or methyl benzoate. The substances should be used in the approximate ratio of one  $C_6H_5$  group in the phenol and in the phenolic compound, to one  $CH_2$  group in the methylene compound. During the reaction ammonia is evolved, and the mixture gradually becomes viscous and solidifies on cooling. At this stage the compound is soluble in alcohol and some other solvents, and may be used as a varnish, etc. The reaction may be carried on up to this stage by boiling a solution of the mixture in alcohol or other non-aqueous solvent. Further heating of the compound produces an infusible, insoluble substance, which may be used as a substitute for amber, as an electrical insulator, etc. The liquid mixture, after short treatment, may be used as a binder for substances like carborundum.—B. V. S.

*Cinnabar [not sensitive to light]; Manufacture of*——  
A. Eibner, Munich, Germany. U.S. Pat. 1,137,467, April 27, 1915. Date of appl., Sept. 4, 1914.

ORES containing mercuric sulphide are treated with a solution of potassium sulphide so as to produce a solution of the double salt,  $HgS.K_2S.5H_2O$ , and this is introduced into a cold solution (saturated with sulphur) of liver of sulphur and flowers of sulphur, whereby black mercuric sulphide and complex potassium sulphides are formed. The entire mass is then heated until cinnabar of the desired tint is obtained.—F. SODN.

*Plastic material.* F. G. Wiechmann, New York. Assignor to Fenoform Corporation, Hastings-upon-Hudson, N.Y. U.S. Pat. 1,135,340, April 13, 1915. Date of appl., Nov. 12, 1909.

THE plastic material is a practically insoluble and infusible substance obtained by the action of a condensation product derived from phenol and formaldehyde, or other suitable compound, upon vegetable protein or vegetable ivory.—E. W. L.

*Rosin and turpentine; Process of removing*——  
from resinous woods. W. M. Bashlin, Grove City, Pa. U.S. Pat. 1,136,994, April 27, 1915. Date of appl., May 11, 1912. Renewed Feb. 10, 1915.

RESINOUS wood is heated under reduced pressure, and is then extracted, un submerged, with a solvent, which removes rosin and turpentine. The extract is removed, and steam is admitted upwards to drive off the last traces of turpentine; the residue is treated with steam passing downwards and with water at about boiling-point, and the products thus separated from the wood are drawn off at the surface of the water.—E. W. L.

*Coumarone-resin from heavy benzols of b. pt. 160°—180° C.; Preparation of a pure, pale*——  
M. Wendriner. Ger. Pat. 281,432, Nov. 8, 1913. Addition to Ger. Pat. 270,993.

THE treatment with sulphuric acid of 60° B. (sp. gr. 1.71), specified in the chief patent (this J., 1914, 474), is omitted, and the benzol is cooled during the addition of the concentrated sulphuric acid, or the latter is added more slowly. Precautions must be taken to avoid overheating of the resin when distilling off the solvent naphtha.—A. S.

*Vinyl esters; Manufacture of useful products [celluloid substitutes, lacquers, etc.] from*——  
Chem. Fabr. Griesheim-Elektron. Ger. Pats. (A) 281,687 and (B) 281,688, July 4, 1913, and April 2, 1914.

(A). VINYL esters, e.g., the acetate or monochloroacetate, are polymerised by exposure to light or by heat. Other substances may be added to the esters before polymerisation, and the products may be softened by heating or dissolved in solvents and subsequently again converted into the solid form. The polymerised esters are non-inflammable, odourless, colourless substances suitable as substitutes for celluloid and similar products, and as insulating materials. Their solutions may be used as lacquers, for impregnating purposes, and for the production of films or threads. (B) Polymerisation is effected in the presence of catalysts, such as organic peroxides or ozonides, or organic acid anhydrides in conjunction with perborates, percarbonates, or certain metallic oxides capable of yielding oxygen. The polymerisation is accelerated, and in certain cases the properties of the products are modified.—A. S.

*Plastic material suitable for the preparation of a horn substitute, films, threads, lacquers, etc.; Production of a*——  
Chem. Fabr. Griesheim-Elektron. Ger. Pat. 281,877, July 4, 1913.

THE products obtained by polymerisation of vinyl halides, with or without the addition of other



substances, are softened or dissolved by means of suitable solvents, and subsequently reconverted into the solid form. The products are non-inflammable and odourless and may be used for all purposes for which cellulose esters or celluloid have been employed hitherto.—A. S.

*Varnish, enamel, or lacquer composition.* J. W. Aylsworth, East Orange, Assignor to Condensite Co. of America, Glen Ridge, N.J. U.S. Pat. 1,137,374, April 27, 1915. Date of appl., Dec. 2, 1913.

SEE Eng. Pat. 3497 of 1911; this J., 1912, 347.

*Preparation of a substitute for boiled linseed oil.* Ger. Pat. 281,452. See XII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

##### PATENTS.

*Rubber substance [rubber sponge] and process of making the same.* P. Schidrowitz and H. A. Goldsbrough, London. Eng. Pat. 1111, Jan. 15, 1914.

RUBBER latex is coagulated under conditions producing a porous or spongy coagulum, and the porous structure is fixed by vulcanisation. Coagulation may be effected by means of a coagulant, or by heat, or by the use of a rubber solvent or precipitant, or by a combination of these. The natural pore formation can be modified or increased by the addition of substances, e.g., carbonates or sulphides, or ammonia, producing gas on subjecting to heat or chemical action. Vulcanisation is effected by means of sulphur, preferably in the presence of a sulphide as carrier, these ingredients being added either before or during the incipient stage of coagulation, after which heat is applied. Fillers, pigments, etc., may be added at any suitable stage. Examples:—(1) Equal volumes of latex and a saturated solution of ammonium carbonate are mixed and heated on a water-bath, and incorporated with 1% of finely divided sulphur; acetic acid is then stirred in, and the containing vessel is placed in a steam vulcaniser and heated for 1 hour at 286° F. (140° C.). (2) 100 grms. of powdered ammonium carbonate is added to 100 c.c. of latex, and the mixture treated with 40 c.c. of carbon bisulphide containing 2 grms. of sulphur. The coagulum is vulcanised under water for about 40 minutes. (3) To 100 c.c. of latex sufficient 5% acetic acid is added to induce creaming; 50 c.c. of water is then added, 3 grms. of finely divided sulphur is stirred in, and sufficient 5% acetic acid added to produce a stiffening, but not complete coagulation. The mixture is vulcanised, with or without previous heating on the water-bath.—E. W. L.

*Rubber; Process of vulcanising*—. A. Cockburn, Edinburgh. Eng. Pat. 13,571, June 4, 1914.

IN vulcanising massive rubber articles, such as solid tyres, a non-porous product is obtained by applying a pressure of about one ton per sq. in. uniformly over the surface of the article, through the intermediary of a liquid, filling, for example, the annular space between a steel or iron mould and the article. The article is wrapped in a suitable flexible covering before being placed in the mould.—E. W. L.

*Purification of unsaturated hydrocarbons containing the conjugated double bond.* Eng. Pat. 6897. See XX.

#### XV.—LEATHER; BONE; HORN; GLUE.

*Hides; Disinfection of — from anthrax spores.* F. W. Tilley. J. Agric. Research, 1915, 4, 65—62.

THE Seymour-Jones method of disinfecting hides with mercuric chloride solution (1 : 5000) and 1% of formic acid (compare this J., 1912, 35), was found to be ineffective, even without neutralisation of the disinfectant; but if the strength of chloride be 1 : 2500, and the treated hides are not exposed for a week or two to any neutralising agent, its employment can be advised. The Schattenfroh process of treatment with a 2% solution of hydrochloric acid containing 10% of salt, followed by 48 hours' exposure (see this J., 1911, 969), gave entirely good results, but Sevcik (Z. Infektionskrankh., 1913, 13, 323—348, 439—452) has found that it did not completely sterilise thick and heavily infected hides. This method, if not perfect, is the best extant. Although more chloride is found in leathers made from hides disinfected by the above processes, the available evidence shows that neither hide nor leather is injured by the disinfection.—E. H. T.

##### PATENTS.

*Leather waste; Compositions utilising*—. J. S. Campbell, London. Eng. Pat. 9370, April 15, 1914.

POWDER obtained by grinding leather scrap is heated with animal fat and with oil (such as fish-oil) and subsequently mixed with hair, wool, or vegetable fibrous material, powdered filling material, and a resilient vulcanisable binder. The mixture may be vulcanised after the addition of some material such as sulphur, or may be softened or liquefied by a suitable solvent and applied to a canvas backing.—F. C. T.

*Patent-leather; Drying of*—. H. Schulz, Worms, Germany. U.S. Pat. 1,137,671, April 27, 1915. Date of appl., March 26, 1915.

PATENT LEATHER is first dried in an oven and then subjected to the simultaneous action of light and ammonia vapour.—E. W. L.

*Leather, artificial; Process for waterproofing and graining*—. C. Krug and H. Böllert. Ger. Pat. 281,302, April 22, 1914. Addition to Ger. Pat. 276,553.

THE process described in the chief patent (see Fr. Pat. 464,842; this J., 1914, 550) may be applied to artificial leather, paper, textile fabrics, etc.—A. S.

*Gelatin; Method for manufacturing sheets of*—. R. A. McQuitty, London. Eng. Pat. 15,465, June 27, 1914.

THIN sheets up to 180 ft. in length are made by spreading liquid gelatin over a flexible band of glazed or japanned material, winding the band round a drying wheel and drying by warm air. The use of ox-gall, oil, or talc for stripping is avoided by this process.—F. C. T.

*Production of a plastic material suitable for the preparation of a horn substitute, films, threads, lacquers, etc.* Ger. Pat. 281,877. See XIII.

#### XVI.—SOILS; FERTILISERS.

*Kelps of the Pacific coast (U.S.A.); Availability of the nitrogen in*—. G. R. Stewart. J. Agric. Research, 1915, 4, 21—38.

THE nitrogen of *Macrocystis pyrifera*, the kelp of most commercial importance, is not in a readily available form, particularly when the kelp is well dried before grinding; the kelp should be

applied in a fresh or partially dried condition. The removal by leaching of the large amount of mineral salts present in both fresh and dried materials, does not affect the availability of the nitrogen. The addition of macrocystis to a fresh soil decreased the rate of transformation of the nitrogen for a time, but the effect was only temporary. The nitrogen of *Nereocystis luelkeana* is much more readily available, and that of *Pelagophycus porra* is available to a moderate extent; but these seaweeds are only of minor commercial importance. Compared with dried blood and cottonseed meal, the fertilising value of the three kelps is very low. The method employed for determining the availability was to trace the extent of ammonification and nitrification taking place in fresh soils of various kinds treated with the fertilisers, under laboratory conditions.—E. H. T.

*Nitrogenous fertilisers; New experiments with — in Germany and Austria.* I.—M. Gerlach, W. Krüger, H. Roemer, B. Tacke, B. Schulze, W. Schneidewind, and H. Immendorff. Ber. über Landwirt., Berlin, 1914. Part 34, 229 pages. II.—O. Reitmair and T. Alexander. Z. Landwirt. Versuchsw. Oesterr., 1914, 17, 729–807. III.—L. Hiltner and F. Lang. Prakt. Blatt. Pflanzenbau u. Pflanzenschutz, 1914, 12, 121–128. Bull. Bureau Agric. Intell., 1915, 6, 212–217. (See also this J., 1914, 1022.)

I.—EXPERIMENTS to determine the value of sodium nitrate, ammonium sulphate, calcium cyanamide, calcium nitrate, calcium nitrite (in a few cases), and liquid manure, were conducted under State auspices at seven stations, of which six (Bernburg, Bremen, Bromberg, Breslau, Halle, Jena) issued reports. The fertilisers were tried on cereal and root crops, growing on sandy, loam, and moor soil, the duration being three years, reduced in some cases to two or one. Taking the value of sodium nitrate as 100, the average values of the others, in the order named, were 84, 76, 99, 90, 64. Ammonium sulphate had a value of only 54 upon the wheat plots, but by neglecting these results the average would be raised to 89. Its effects on peaty soils were remarkable: viz., rye 109, oats 103, potatoes 102, and it gave better results on sandy than on loam soils, especially with root crops. There was no considerable difference between the effects of cyanamide on sandy and on loam soils. The various nitrogenous fertilisers had little influence on the percentage of nitrogen in the crops. II.—A comparison of the effects of calcium and sodium nitrates was carried out on similar lines by the Vienna Agric. Exper. Stat. Of 188 experiments, 91 gave reliable and 69 uncertain results, the rest being failures. In general, the action of sodium nitrate was better than that of calcium nitrate, especially on root crops. III.—A series of comparative experiments with cyanamide, etc., on barley and potatoes, grown on light soil at Moosacher, Bavaria, showed that cyanamide is valuable particularly as a top-dressing, for barley, but is not to be recommended for potatoes.—E. H. T.

*Soil moisture; Effect on — of changes in the surface tension of the soil solution brought about by the addition of soluble salts.* P. E. Karraker. J. Agric. Research, 1915, 4, 187–192.

THE results of preliminary experiments indicate that changes in the surface tension of the soil solution arising from the application of fertiliser salts are of no importance in affecting the moisture condition of the soil.

*Nitrogen transformation in soils; Antagonism between anions as related to —.* C. B. Lipman. Plant World, 1914, 17, 295–305. Bull. Bureau Agric. Intell., 1915, 6, 205.

SOLUTIONS of sodium chloride, sulphate, and car-

bonate show a mutual antagonism as regards their influence on ammonification in culture experiments. This is the case not only when the stimulating carbonate is one of the salts employed, but also when the toxic sulphate and chloride are used together. Nitrification is depressed in presence of 0.2% of sodium chloride, but increased by 0.05–0.2% of the sulphate. It is depressed 75% below the normal by 0.05% of the carbonate, but the addition of sulphate up to 0.5% then increases it above the normal. When chloride and carbonate are applied together, nitrification is increased by more than 25% above the normal; and the inhibiting action of carbonate present in large quantities can be neutralised, and a stimulating effect obtained, if 0.2% of chloride be added.

—E. H. T.

*Ammonia from nitrogenous organic substances; Influence of sugars upon the [bacteriological] formation of —.* E. Auel and H. Colin. Comptes rend. Soc. Biol., 1914, 76, 835–837. Bull. Soc. Chim., 1915, 17, 140.

WHEN cultivated upon an entirely nitrogenous medium, *Micrococcus prodigiosus*, Kiehl's bacillus, *Bacillus violaceus*, Eberth's bacillus, and *Proteus vulgaris* always give rise to the formation of a large amount of ammonia; but if dextrose be added, the medium becomes acid. It would therefore appear that the above micro-organisms effecting ammonification no longer behave in their characteristic manner towards nitrogenous substances when assimilable carbohydrates are available.

—J. P. O.

#### PATENTS.

*Fertiliser containing lime nitrogen and tankage material.* F. S. Washburn, Nashville, Tenn. U.S. Pat. 1,135,639, April 13, 1915. Date of appl., Nov. 17, 1914.

COMMERCIAL calcium cyanamide, or lime nitrogen, is intimately mixed in predetermined proportions with tankage fertiliser material containing phosphoric compounds, so as to yield a dry product.

—H. R. D.

*Phosphates; Manufacture of soluble —.* W. B. Bottomley, London. Eng. Pat. 20,788, Oct. 9, 1914.

FINELY divided mineral phosphate is moistened with a putrefying liquor containing a small proportion of a suitable food for micro-organisms, e.g., gelatin or meat extract, together with aerobic organisms, and the mass is maintained at about 30° C. for about a week.—B. N.

*Phosphoric acid; Process of rendering available —.* W. S. Landis, Niagara Falls, N.Y., Assignor to F. S. Washburn, Nashville, Tenn. U.S. Pat. 1,137,065, April 27, 1915. Date of appl., March 3, 1915.

FINELY powdered phosphate rock is mixed with sodium sulphate and carbon, the mixture is heated to eliminate the acid radical from the salt, then heated more strongly to cause incipient fusing and clinkering, discharged from the furnace, and ground.—B. N.

*Acid phosphates [superphosphates] and the like; Manufacturing and curing —.* G. L. Pratt, Atlanta, Ga. U.S. Pat. 1,137,531, April 27, 1915. Date of appl., Jan. 30, 1912.

THE finely powdered phosphatic mass is mixed with acid so as to convert it into a viscous mass, which is discharged into an underlying curing den to effect an initial curing, and the resulting dry and friable mass is discharged into an underlying carrier. The latter is transported on a substantially horizontal plane, and the contents are dis-

charged into an underlying storage place for the final curing operation. The various discharging operations from a higher to a lower plane prevent rubbing pressure on the friable mass, which would convert it into a sticky condition.—B. N.

## XVII.—SUGARS; STARCHES; GUMS.

*Sugar cane; Deterioration of cut*—W. E. Cross and J. A. Belile. Intern. Sugar J., 1915, 17, 218—224.

EXPERIMENTS by Hall (Revista Ind. de Tucumán, 1913, 4, 148) have proved that the deterioration of cut cane is not governed by the size of the cane, the thickness of the rind, infestation by borers, or other such conditions. The authors' experiments indicate that the deterioration is due to the presence of enzymes. Stored canes were crushed, one portion (A) of the resulting juice was analysed, and another portion (B) was treated with a mixture of chloroform and toluene (to exclude the action of bacteria), and preserved for 17 days, when it was found that the glucose ratio of (B) had increased very considerably over that of (A). Freshly-milled cane showed similar results under identical conditions, though to a less marked degree, this pointing to the fact that the enzyme is elaborated during storage. The action of invertase in cut cane appears to be connected with the preparation on the part of the plant for germination, the sucrose being hydrolysed to the more readily assimilable invert sugar for the use of the young plant. This would account for the relative susceptibility to inversion of different varieties; the more rapidly germinating kinds invert more rapidly when stored after cutting. In practice deterioration may be prevented or lessened by storing the cane in a heap, covering with trash, and, preferably, drenching with water at frequent intervals.—J. P. O.

*Bagasse; Determination of sugar in*—N. Deerr. Intern. Sugar J., 1915, 17, 213—215.

LOOSELY-PACKED bagasse (495 grms.) contained in a metal basket (5½ in. wide × 10½ in. high) is placed in an extractor consisting of a cylinder (6 in. wide × 12½ in. high) filled with hot water (containing a little sodium carbonate) up to a draw-off cock 8½ in. from the bottom, and fitted with a reflux condenser. The extractor is placed on an electric stove, the contents are boiled for an hour, then cooled below boiling point, mixed, and 1027 c.c. of the liquid is drawn off and evaporated to slightly less than 200 c.c., preferably on an electric stove provided with a low-water alarm. After cooling, making up the volume to 200 c.c. with basic lead acetate, and filtering, the saccharimetric observation is made, using a 400 mm. tube, the result being multiplied by 0.2 to give the polarisation of the bagasse. By using a large sample, the tedious processes of cutting and sub-sampling are obviated, and by concentrating the dilute aqueous extract a reading of sufficient magnitude to eliminate the error of observation is obtained.—J. P. O.

*Sugar; Detection of small quantities of*—by the production of formaldehyde, and the constituents of urine which give rise to formaldehyde. E. Sal-kowski. Z. physiol. Chem., 1915, 83, 432—446.

If a dilute solution of formaldehyde (e.g., 1 part in 50,000) is treated with a small quantity of Witte's peptone, and after the peptone has been dissolved by shaking and warming, the solution is treated with 3 drops of 3% ferric chloride solution, and then heated to boiling with about half its volume of hydrochloric acid of sp. gr. 1.19, it becomes violet and finally deep blue, and this colour remains unchanged for months. Formaldehyde is produced in the oxidation of sugar by acid solutions of potassium permanganate (cp. Rosenthaler, this

J., 1914, 224), and the author has endeavoured to apply this reaction, coupled with the colour test for formaldehyde given above, to the detection of sugar in urine. The oxidising solution was made by mixing 30 c.c. of dilute sulphuric acid (200 grms. diluted to 1 litre), 20 c.c. of 1%, or in some cases 5%, potassium permanganate solution, and 50 c.c. of water. The efficacy of the mixture diminishes on long storage, manganese dioxide being deposited, but a slight precipitate does not interfere with the reaction. When the stronger permanganate solution is employed the mixture is best kept in a loosely stoppered bottle as it slowly evolves oxygen. Two c.c. of a 0.1% solution of dextrose was heated with 5 c.c. of this mixture (made with 1% permanganate solution) until manganese dioxide began to separate; after decolorisation by a few drops of oxalic acid solution the liquid showed a strong formaldehyde reaction. Two c.c. of a 0.02% dextrose solution treated in the same way gave no reaction for formaldehyde, probably owing to further oxidation, for when 2—2.5 c.c. of the oxidising mixture was used instead of 5 c.c., a strong formaldehyde reaction was obtained. Two c.c. of normal urine, oxidised with 5 c.c. of the permanganate mixture as described above, and tested for formaldehyde, gave an intense reaction. Attempts to remove the substances responsible for this production of formaldehyde, were unsuccessful, so resource was had to precipitation of the sugar by Brücke's reagent—basic lead acetate in presence of ammonia. It was found that 0.2% of dextrose in urine can be detected with certainty if a measured quantity of the urine is precipitated first with basic lead acetate, and after filtration, with ammonia and basic lead acetate, and the well washed second precipitate is dissolved in hot acetic acid, the solution made up to the original volume of the urine, and 2 c.c. of it oxidised with 5 c.c. of permanganate mixture and tested for formaldehyde. Creatinine produces formaldehyde on oxidation, but the quantity of the former retained by the Brücke precipitate is not sufficient to interfere with the test, though it might exert a reducing action on Fehling's solution.—J. H. L.

*Sucrose; Action of chloroform upon the inversion of*—in the beetroot. P. Mazé. Comptes rend. Soc. Biol., 1914, 77, 549—550. Bull. Soc. Chim., 1915, 17, 143.

ON placing slices of beetroot containing about 7.5% of sucrose in solutions of chloroform or ether, it was observed that these reagents exerted an accelerating effect upon the action of the invertase, so that a greater formation of reducing sugars took place than occurred when the slices were immersed in pure water.—J. P. O.

*Sucrose solutions; Vapour pressure of concentrated*—D. O. Wood. Faraday Soc., May 11, 1915. [Advance proof.] pp. 19.

THE accompanying table shows the vapour pressure of sucrose solutions, containing 47.97, 61.02, and 69.20 grms. of sugar per 100 grms. of

47.97% solution.		61.02% solution.		69.20% solution.	
°C.	Pressure.	°C.	Pressure.	°C.	Pressure.
60.42	14.80	62.98	15.18	59.0	11.27
63.39	16.58	67.51	18.69	62.9	13.60
66.02	18.47	73.08	23.75	68.2	17.37
72.95	24.78	78.55	29.86	73.9	22.25
74.23	26.25	83.35	36.38	75.1	23.50
80.42	34.49	87.61	42.93	78.8	27.68
82.73	37.39	90.85	48.58	84.9	35.04
84.86	40.10	[92.66]	52.71	89.1	41.84
90.00	49.60	—	—	92.0	46.44

solution, in cm. of mercury, measured directly by a static method. The bearing of the results on the theory of solutions is discussed. For the second and third solutions the lowering of vapour pressure due to the sugar, expressed as a fraction of the vapour pressure of water, diminishes slightly with rise of temperature, indicating a negative latent heat of dilution and a slight apparent fall in the osmotic pressure with rise of temperature. Application of Callendar's hydration formula indicates the association of four molecules of water with each molecule of sugar.—J. H. L.

*Glucose [dextrose]; Derivatives of a new form of —.* J. C. Irvine, A. W. Fyfe, and T. P. Hogg. Chem. Soc. Trans., 1915, 107, 524–541.

DEXTROSE when shaken for some hours at ordinary temperatures with methyl alcohol containing 1% HCl, is converted largely into a glucoside designated by Fischer as  $\gamma$ -methylglucoside (this J., 1914, 770). This new glucoside is characterised by the ease with which it is hydrolysed by acids, resembling in this respect sucrose and the fructosides, by its capacity to reduce potassium permanganate solution, and by its tendency to unite with an atom of oxygen to give a neutral substance which readily undergoes auto-condensation to a product allied to the disaccharides. It is converted on methylation into tetramethyl- $\gamma$ -methylglucoside, b. pt. 106° C. at 0.25 mm.,  $[\alpha]_D$  in water =  $-14.6^\circ$ ,  $n_D = 1.4453$ . A study of the hydrolysis of this tetramethyl-derivative to tetramethyl- $\gamma$ -glucose, which takes place easily with N/100-hydrochloric acid at 40° C., showed that it is a mixture of isomeric compounds. Tetramethyl- $\gamma$ -glucose retains the capacity to reduce permanganate, and is distinguished from normal methylated glucoses, in chemical reactivity and in all its optical properties. The freshly distilled substance exhibits mutarotation, developing an increase in laevorotatory power. It gives no osazone, but on reduction is converted into a tetramethylhexitol; from its oxidation products a tetramethylgluconic acid was isolated and identified by conversion into the lactone.—G. F. M.

*Re-burning of lime from alkali waste and other forms of precipitated carbonate of lime.* Meade. See VII.

*The sugars of dormant and germinating barley and wheat.* Kluyver. See XVIII.

#### PATENT.

*Process of treating sugar cane.* [Utilisation of bagasse for paper making.] Eng. Pat. 17,834. See V.

### XVIII.—FERMENTATION INDUSTRIES.

*Barley and wheat; The sugars of dormant and germinating —.* A. J. Kluyver. Biochem. Suikerbeepal. Brewers' J., 1915, 51, 289–290.

ABOUT 60 grms. of finely ground cereal was digested for 5 hours at 35° C. with 200 c.c. of 80% alcohol and a little calcium carbonate; 130 c.c. of the filtrate was concentrated and the residue was dissolved in 10 c.c. of water. The solutions were fermented, using van Iterson's apparatus, by means of six yeasts of known fermentative power for different sugars, viz.: *S. cerevisiae* (top and bottom), *Torula dattila*, *T. monosa*, *Schizosacch. Pombe*, and lactose yeast. From the results, set forth in the tables, it would appear that maltose, which is absent from the dormant corn, is produced during germination. The sucrose of barley is partially removed during steeping but increases during germination; the raffinose disappears but the monoses (dextrose

and levulose) increase, although a slight destruction of these takes place during kilning.

#### Percentage of sugars in wheat.

Sugars.	Dormant.	After germinating.		
		44 hours, corns chitted.	9 hours, rootlets 0.5–1.0 cm. long.	12 hours, rootlets 3 cm. and over.
Raffinose ..	0.08	—	—	—
Sucrose ....	0.73	0.08	1.53	—
Monoses ..	0.09	0.20	0.43	2.79
Maltose ....	—	0.31	1.03	3.19

#### Percentage of sugars in barley.

Sugars.	Dormant.	After 24 hours.	After steeping 48 hours and germinating.					After curing.	
			24 hrs.	72 hrs.	96 hrs.	120 hrs.	144 hrs.	24 hrs. at 38°–50° C.	48 hrs. at 38°–50° C.
Raffinose	0.45	0.36	0.43	—	—	—	—	—	—
Sucrose ..	0.76	0.56	0.67	0.54	2.14	3.01	3.01	4.44	4.29
Monoses	0.04	0.11	0.10	0.23	1.01	1.43	1.46	1.48	1.33
Maltose	—	—	—	0.47	1.91	2.41	2.84	0.52	1.03

—J. F. B.

*Amylases; Studies on —. X. Comparison of certain properties of pancreatic and malt amylase preparations.* H. C. Sherman and M. D. Schlinger. J. Amer. Chem. Soc., 1915, 37, 1305–1319.

THE purified amylase preparations obtained from pancreas and from malt are similar in many respects but are not identical substances. Both contain 15–16% N and give similar group-reactions in the chemical classification of protein substances. When heated in solution both yield coagulated albumin and a proteose or peptone in solution; the biuret reactions of these filtrates are pinker than those of the original enzymes. While malt diastase exerts its optimum diastatic power in a slightly acid solution (see this J., 1915, 37(1), the optimum medium for the action of pancreatic amylase is slightly alkaline, expressed in Sørensen's terms by  $p_H^+ = 8-8.5$ . When each amylase was allowed to act on soluble starch for 30 mins. at 40° C. under its optimum conditions of reaction and salt concentration, the pancreatic amylase showed a diastatic power of 4000 on the "new scale" or 6000 Lintner, producing 10,000 times its own weight of maltose, while malt amylase showed 1570 "new scale" or 2350 Lintner, corresponding to 4000 parts of maltose. Similar differences were observed in long digestion experiments. The most highly purified specimens of pancreatic amylase possess a pronounced proteolytic action on casein and gelatin, whereas purified malt diastase is devoid of proteolytic activity. In pancreatic amylase the amylolytic and saccharogenic powers are concentrated in practically the same ratio, whereas in purified malt amylase these activities are divergent. Both enzymes deteriorate on standing in dilute aqueous solution, but malt amylase is always more stable than the pancreatic. On the other hand, the pancreatic amylase shows greater stability in 50% alcoholic solution and shows a more sustained activity in long-period digestions with an excess of substratum and suitable concentration of electrolytes. Both amylases deteriorate during dialysis, much more rapidly at the ordinary temperature than at 5°–10° C.—J. F. B.



treated by preliminary acidification of the mash. The subsidiary acid mash should be added when the main mash has reached  $140^{\circ}$ – $143^{\circ}$  F. ( $60^{\circ}$ – $62^{\circ}$  C.): this eliminates bottom yeast cells, and also facilitates clarification of raw grain worts and improves the stability of the beer. For a full-bodied beer the most valuable dextrins are not those produced by slow saccharification of raw grain below the final mashing temperature, but those produced between  $149^{\circ}$  and  $158^{\circ}$  F. ( $85^{\circ}$ – $70^{\circ}$  C.). It is most important to boil sharply either with direct fire or high-pressure steam; in the latter case a coil in the mash is preferable to a double-bottomed copper in order to produce a greater caramelising effect. The hops should be added after the wort has boiled for 15–30 mins.; slow passage through the cooler is advisable. A high temperature of fermentation favours the production of acids, which may be so pronounced as to detract from the full-bodied character of the beer, but there is less danger when the malt employed is not too mellow. Contact with air operates against fullness of body, and open fermentation vessels often give poor results with beers of low gravity. A good proportion of carbon dioxide in the beer is indispensable for the full-bodied character. In applying the above rules, attention must be paid to the preservation of stability, and fullness of body must not depend on the presence of traces of starch; the employment of fresh hops in sufficient proportions should always be maintained.—J. F. B.

*Methyl alcohol; Determination of*—in presence of ethyl alcohol. G. C. Jones. Analyst, 1915, 40, 218–221.

SIMMONDS' directions (this J., 1912, 150) for carrying out Denigès' method for the determination of methyl alcohol need slight amendment in order to obtain accurate results. The Schiff's reagent employed should be prepared by dissolving 0.2 gm. of magenta in 10 c.c. of saturated sulphur dioxide solution and, after 24 hours, diluting the solution with water to 200 c.c. The amount of permanganate solution used for the oxidation must not be less than 2.5 c.c.; when 5 c.c., is employed the sensitiveness of the test is doubled. The quantity of sulphuric acid added with the permanganate may vary from 0.1 to 0.3 c.c., and the period of oxidation, provided it be not less than 3 minutes, appears to be without effect on the results. The subsequent addition of sulphuric acid must be nearly the same in amount in any one set of experiments. Five c.c. of the Schiff's reagent should be added in each case; if 10 c.c. is added, the acid concentration is so much reduced that acetaldehyde from ethyl alcohol develops a coloration. The test may be expected to show the presence of about 0.1% of methyl alcohol in ethyl alcohol, and proportions of about 1% can be determined with an error not exceeding 5%.

—W. P. S.

*Alcohol from cassava.* A. E. Collens. Bull. Dept. of Agric., Trinidad and Tobago, 1914, 14, 56.

SWEET cassava roots, when pulped, boiled, saccharified with malt, fermented, and distilled, yielded a quantity of alcohol corresponding to 75.6 galls. of 91% alcohol per ton of dry material. In another experiment, where the starch was saccharified with taka-diastase, the yield amounted to 81.5 galls. of 94% alcohol per ton of air-dried slices.

—W. P. S.

#### PATENTS.

*Fermenting tuns.* F. Rogerson, London. Eng. Pat. 22,820, Nov. 20, 1914.

THE interior surface of ferro-concrete tuns is coated first with about  $\frac{1}{2}$  in. of damp-proof cement (e.g., cement mixed with soapy or greasy materials) and

then with about  $\frac{1}{2}$  in. of a porous mixture such as 3 parts of sand and 1 part of cement. When this is dry it is heated and covered with a layer, about  $\frac{1}{2}$  in. thick, of a waterproof and acid-resistant mixture of waxes, e.g., beeswax 60%, ceresin 20%, paraffin wax 10%, and carnauba wax 10%, with or without a gum or resin.—J. H. L.

*Beer; Process of charging*—O. M. Lamsens, Detroit. U.S. Pat. 1,137,149, April 27, 1915. Date of appl., May 20, 1912.

SEE Fr. Pat. 457,789 of 1913; this J., 1913, 1026.

*Process for making a decoloriser [from argol].* U.S. Pat. 1,135,216. See IIb.

#### XIXA.—FOODS.

*Wheat gluten; Colloidal swelling of*—F. W. Upson and J. W. Calvin. J. Amer. Chem. Soc., 1915, 37, 1295–1304.

WHEAT gluten, washed in a stream of distilled water until free from starch, was pressed to a cake from which discs of uniform size and weight were cut, weighed, steeped in various solutions, drained, and weighed again. When moist gluten is steeped in a solution of any acid it swells and absorbs water to an amount depending on the nature and the concentration of the acid. Maximum absorptions are recorded for 0.005N hydrochloric acid, lactic acid between 0.01 and 0.02N, and 0.04N acetic acid. With increase in the concentration of acid the absorption of water rapidly decreases, and in 0.2–0.5N hydrochloric acid the moist gluten loses water. The absorption of water by, and its withdrawal from, the gluten discs is a reversible process which may be performed on the same disc within wide limits without substantial change. The presence of neutral salts opposes the absorption of water by gluten in acid solutions, and, with higher concentrations of salt, may even cause loss of water from moist gluten. Gluten which has taken up water in an acid solution loses water and regains its original properties when placed in a salt solution. Non-electrolytes, such as glycerol and flour or bran extracts, also oppose the absorption of water but in a less marked degree than salt solutions; sugar has very little action except in high concentrations. The absorption of water in acid solutions is increased by rise of temperature. In all these respects the behaviour of gluten is analogous to that of animal colloids such as gelatin, etc.

—J. F. B.

*Milk; Human*—A. W. Bosworth. J. Biol. Chem., 1915, 20, 707–709.

HUMAN milk was filtered through a clay filter and the serum and unfilterable portion analysed separately (cf. this J., 1915, 297); the results obtained suggest the following arrangement as representing the condition in which the constituents are present in human milk of normal composition:—Fat, 3.30; lactose, 6.50; proteins combined with calcium, 1.50; calcium chloride, 0.059; monopotassium phosphate,  $(\text{KH}_2\text{PO}_4)$ , 0.069; sodium citrate, 0.055; potassium citrate, 0.103; monomagnesium phosphate,  $(\text{MgH}_2\text{P}_2\text{O}_7)$ , 0.027%.—W. P. S.

*Fat [in animal substances, etc.]; Determination of*—H. Rosenthal and P. F. Trowbridge. J. Biol. Chem., 1915, 20, 711–717.

THE sample is saponified with sodium hydroxide, then diluted with water, acidified with hydrochloric acid, and shaken with ether. The ethereal solution is evaporated to dryness, the residue dissolved in a mixture of petroleum spirit and alcohol, and the solution titrated with N/20 sodium hydroxide solution, using phenolphthalein

as indicator; the neutralised solution is evaporated and the residue dried to constant weight. After deducting the weight of alkali present, the residue of fatty acids is calculated to glyceride, the weight of fatty acid being multiplied by 1.046 for this purpose. The results obtained are more concordant than those yielded by the Kumagawa-Suto method (this J., 1908, 455). To determine fat in blood, an extraction with alcohol should first be made, as recommended by Shimidzu (this J., 1910, 1259).—W. P. S.

*Wheat bran; Organic phosphoric acid compound of —. IV. Occurrence of inositol triphosphate in wheat bran.* R. J. Anderson. J. Biol. Chem., 1915, 20, 463—473. (See also this J., 1914, 936.)

THE insoluble barium salts obtained by the addition of barium hydroxide to the aqueous hydrochloric acid extract of wheat bran, were further separated by converting into acid barium salts and extracting with cold water. The insoluble portion gave a crystalline strychnine salt forming either needles or plates, which were apparently homogeneous, and which on re-conversion into the barium salt gave a white amorphous compound of the composition,  $C_6H_9O_{15}P_3Ba_2$ , barium inositol-triphosphate. Precipitation of the hydrochloric acid solution of this salt with alcohol furnished an acid salt of the composition,  $(C_6H_9O_{15}P_3)_2H_2Ba_3$ . The free inositol triphosphate,  $C_6H_9O_{15}P_3$ , prepared from the barium salt, formed a colourless syrup, soluble in water and in alcohol. Unlike phytic acid it gives no precipitate with ammonium molybdate or silver nitrate, and does not at once precipitate egg albumin. The acid does not give a precipitate with barium or calcium chlorides unless previously neutralised with ammonia. On hydrolysis with dilute sulphuric acid at  $150^\circ C$ , it is decomposed into inositol and phosphoric acid.—G. F. M.

*Phytin; Hydrolysis of — by the enzyme, phytase, contained in wheat bran.* R. J. Anderson. J. Biol. Chem., 1915, 20, 475—482.

THE chief products of the hydrolysis of phytin by the phytase contained in wheat bran are phosphoric acid and intermediate products consisting of inositol tri-, di-, and monophosphate, identical with the compounds contained in 0.2% hydrochloric acid extracts of the bran. The whole of the phytin is at least partially hydrolysed, and a portion of it completely, since the resulting solution contains free inositol.—G. F. M.

*Phytase; Hydrolysis of the organic phosphorus compound of wheat bran by the enzyme —.* R. J. Anderson. J. Biol. Chem., 1915, 20, 483—491.

THE presence of phytase in wheat bran causes a rapid hydrolysis of the organic phosphorus compound when the bran is digested with water (see also preceding abstract). The maximum activity of the enzyme occurs in the presence of 0.1% hydrochloric acid and 0.2% acetic acid, and this affords an explanation of the presence of the lower inositol-phosphoric acids, and of the large proportion of inorganic phosphoric acid found in hydrochloric acid extracts of wheat bran. With increasing concentration of hydrochloric acid the activity rapidly diminishes, and by exposure to 0.5% hydrochloric acid the enzyme is destroyed. Wheat bran normally contains about 0.1% of inorganic phosphorus, that is, about 11% of the total soluble phosphorus.—G. F. M.

*Phytin in wheat bran.* R. J. Anderson. J. Biol. Chem., 1915, 20, 493—500.

FROM the extract obtained by digesting wheat bran with 1% hydrochloric acid, which is sufficiently strong to inhibit the hydrolytic action of phytase

(see preceding abstract) crystalline barium salts of the composition:  $C_6H_{11}O_{14}P_3Ba_2 \cdot 8H_2O$  and  $(C_6H_{11}O_{14}P_3)_2Ba_3 \cdot 14H_2O$  were isolated. These salts were identical with the tribarium phytate and heptabarium phytate obtained from oats, corn (maize), commercial phytin, etc., and wheat therefore contains in common with these products the same phosphorus compound, namely, phytic acid or inositol hexa-phosphate.—G. F. M.

*Cheese; Determination of chlorides in —.* E. C. V. Cornish and J. Golding. Analyst, 1915, 40, 197—202.

THE cheese is heated with sulphuric acid in a Kjeldahl flask, into the neck of which is inserted a soda-lime tube of such size that the bulb just blocks the mouth; the evolved gases are aspirated through this tube into silver nitrate solution containing 40% of its volume of nitric acid, and the excess of silver nitrate is then titrated with thiocyanate solution. The method yields concordant results and is more reliable than the ordinary incineration process, owing to the risk of volatilising chlorides in the latter method.—W. P. S.

*Cassava; Hydrocyanic acid content of bitter and sweet —.* A. E. Collens. Bull. Dept. of Agric., Trinidad and Tobago, 1915, 14, 54—56.

THE following percentage quantities of hydrocyanic acid were found in (A) sweet and (B) bitter cassava plants, the determinations being made immediately after the plants had been dug up:—Leaves (A) 0.0162, (B) 0.041; peel of stem, (A) 0.043, (B) 0.113; pith of stem, (A) 0.019, (B) 0.076; edible portion of root, (A) 0.0048, (B) 0.053. The hydrocyanic acid content of different parts of the freshly dug roots was the same, but after keeping for 3 days the upper part of the sweet roots contained about twice as much as the bottom portion; loss of water during air-drying also caused the hydrocyanic acid content to increase. When sweet cassava was boiled with water for 1 hour, or roasted, no hydrocyanic acid was developed.—W. P. S.

*Carbon dioxide in baking powder and carbonates; Determination of —.* H. W. Brubaker. J. Ind. Eng. Chem., 1915, 7, 432—433.

THE baking powder is treated with a saturated solution of sodium chloride, and the carbon dioxide evolved is caused to displace its own volume of a saturated salt solution, in a graduated cylinder. In the case of carbonates the sample is treated first with 10 c.c. of dilute hydrochloric acid saturated with sodium chloride and then, in a second test, with 15 c.c. of the acid solution; from the difference between the two results the correction required for the solubility of carbon dioxide in the acid solution is ascertained. To obviate the necessity of making a correction, 0.5 gm. of precipitated calcium carbonate may be treated with 30—40 c.c. of dilute (1:3) hydrochloric acid, and when effervescence has ceased, 10 c.c. of the clear acid, saturated with carbon dioxide, withdrawn and used in the test.—A. S.

*The sugars of dormant and germinating barley and wheat.* Kluyver. See XVIII.

*Action of erepsin.* Rice. See XVIII.

#### PATENTS.

*Flour; Method of treating —.* F. D. Larabee, Hutchinson, Kansas, U.S.A. Eng. Pat. 18,506, Aug. 10, 1914.

THE flour is passed between disc grinders of metal with the opposing faces suitably ridged. A rubbing action takes place which breaks down the starch cells, so that more water is absorbed when the

flour is made into dough. The treatment may be effected also by the use of rollers working at a low differential speed and capable of exerting a high pressure.—J. H. J.

*Coffee tablet; Process of making a* — R. Hübner, Assignor to G. H. Benjamin, New York. U.S. Pat. 1,137,265, Apr. 27, 1915. Date of appl., Dec. 27, 1909.

COFFEE is extracted with ether, or ether and alcohol, and afterwards with water. The ethereal extract is treated with an alkali and the saponified substances are separated. The two extracts are then mixed and evaporated, and the residue formed into tablets.—J. H. J.

*Food product [from cottonseed oil]*. J. J. Burchenal, Assignor to The Procter and Gamble Co., Cincinnati, Ohio. U.S. Pat. 1,135,351, Apr. 13, 1915. Date of appl., Nov. 10, 1910.

COTTONSEED oil is incompletely hydrogenised so as to produce a lard-like product of a yellowish or white colour, having an iodine value of 55–80, m. pt. 33°–40° C., titer value 42°–35° C., and giving no reaction for cottonseed oil with Halphen's test.—J. H. J.

*Flour; Method of treating* — F. D. Larabee, Hutchinson, Kans., Assignor to W. G. Andrews, Chicago, Ill. U.S. Pat. 1,136,881, April 20, 1915. Date of appl., July 11, 1913.

SEE Eng. Pat. 18,506 of 1914; preceding.

*Nitrogen compounds from organic substances of animal origin; Production of soluble* — E. Bohon, Assignor to J. Sloan, Paris. U.S. Pat. 1,138,348, May 4, 1915. Date of appl., Feb. 20, 1914.

SEE Fr. Pat. 466,150 of 1913; this J., 1914, 661.

*Milk, artificial; Manufacture of* — F. Gössel, Frankfurt, Germany. U.S. Pat. 1,139,031, May 11, 1915. Date of appl., May 5, 1914. Renewed Mar. 25, 1915.

SEE Fr. Pat. 451,447 of 1912; this J., 1913, 621.

## . XIXB.—WATER PURIFICATION; SANITATION.

*Calcium and magnesium carbonates; Solubility of — in waters free from carbonic acid, in reference to their salt content and organic substances*. F. Gothe. Chem.-Zeit., 1915, 39, 305–307, 326–327.

The solubility of calcium carbonate in water free from carbonic acid varies between narrow limits, amounting on the average to 17.4 mgrms. CaO or 31.0 mgrms. CaCO<sub>3</sub> per litre. The solubility of magnesium carbonate under similar conditions is subject to greater variations, being on the average 94.4 mgrms. MgCO<sub>3</sub> per litre. The solubility of both calcium and magnesium carbonates is increased by the chlorides, nitrates, and sulphates of the alkalis and lowered by the carbonates of the alkalis, and the chlorides, nitrates, and sulphates of the alkaline earths. Ammonium salts cause the decomposition of calcium and magnesium carbonates, hence an increase in their solubility as such is not in question. Organic substances alone, and particularly in presence of neutral salts of the alkali metals, retard the decomposition of the bicarbonates of calcium and magnesium, otherwise the solubility relationships of the carbonates in presence of alkali or alkaline-earth salts above mentioned, holds without exception in this case, the reduction in solubility in presence of calcium or magnesium chlorides being particularly marked. —G. F. M.

*Nitrous acid [in water]; Colorimetric determination of* — G. Romijn. Chem. Weekblad, 1914, 11, 1115–1116. Analyst, 1915, 40, 254.

THE reagent used consists of a powdered mixture of *a*-naphthylamine hydrochloride, 1, tartaric acid, 89, and sulphuric acid, 10 parts; it keeps unaltered for a long time. The water under examination must not contain more than 0.15 mgrm. NO<sub>2</sub> per litre, since otherwise precipitation of the colouring matter formed in the reaction takes place. Should the coloration appear too rapidly, the water must be diluted with distilled water and the test repeated.—W. P. S.

*p-Dichlorobenzene; Use of — as insect fumigant*. A. B. Duckett. Bull. U.S. Dept. Agric., No. 187, Feb. 10, 1915. 7 pages.

*p-DICHLOROBENZENE* vapour is heavy, has an ethereal odour, and although harmless to man, animals, plants, and fruits, is fatal to a large variety of insects, both in the adult and larval states. Owing to its great volatility and to the fact that the optimum temperature is 75°–85° F. (24°–30° C.), this fumigant is best applied in a confined space, *e.g.*, in a fumigating chamber, but it can be used in a closed cupboard or a dwelling room. The amount required is from 12–16 oz. per 100 cub. ft. of space, and the time needed is 24–36 hours. It is non-inflammable and very cheap.—E. H. T.

*Quaternary salts of hexamethylenetetramine*. Jacobs and Heidelberger. See XX.

*Reaction between calcium hydroxide and sulphur in aqueous solution [during preparation of lime-sulphur wash]*. Auld. See VII.

*Precipitant for ammonia. (Substitute for Nessler's reagent.)* Graves. See XXIII.

### PATENTS.

*Water, more particularly feed-water for steam generators, hot-water systems, and the like; Purification of* — C. Haythorpe, London. Eng. Pat. 9396, April 16, 1914.

IN filters for purifying water by galvanic action, copper strips are passed through the carbon of the filter and bolted to zinc blocks forming the ends of the filter. The filter is surrounded by a steam jacket, and is placed at the side of the feed-water pipe and connected to it by inlet and outlet valves, between which is a valve on the feed-pipe. When it is desired to clean the filter, the last-mentioned valve is opened and the inlet valve to the filter closed, whereupon the water passes backwards through the filter and escapes through a scum pipe.—J. H. J.

*Sterilisation of liquids [; Electrical —]*. P. H. Dawe, London. Eng. Pat. 9567, April 17, 1914.

IN the sterilisation of liquids by alternating electric currents, the liquid is passed through a narrow electric zone, at a uniform and relatively high electric density, and disposed transversely to the direction of flow of the liquid. The effective surface of the electrodes may be increased without increasing the thickness of the effective zone; *e.g.*, one or both electrodes may be shielded in a chamber or chambers to which the liquid has access, being fixed in such a manner that as the supply of liquid subsides, the electrodes are gradually uncovered. When a static transformer is employed, the supply of current is automatically controlled by a rheostat as the quantity of liquid is varied.—B. N.

*Sewage sludge and the like; Apparatus for drying* — J. Bromet, F. Thorman, and H. C. Wood, Tadcaster, Yorks. Eng. Pat. 14,615, June 18, 1914.

To effect the better breaking up and the more



efficient drying of sewage sludge in the apparatus described in Eng. Pat. 7822 of 1913 (this J., 1914, 565), the heated rotating cylinders are provided, preferably at the feed end, with one or more internal rollers which work against the interior wall of the cylinder and are attached to lever arms hinged to brackets fixed to the girder running through the cylinder. A scraper attached to the girder removes any material adhering to the cylinder. At the exit end of each cylinder a pair of external rollers rotating in opposite directions on fixed and movable shafts, completes the crushing of the material.—J. H. J.

*Sewage and analogous liquids; Apparatus for the purification of*—. W. Jones, and Jones and Attwood, Ltd., Stourbridge. Eng. Pat. 19,916, April 11, 1914.

In an aerating tank for the treatment of sewage, the floor of the tank is divided into a number of saucer-like depressions, and in the centre of each depression is a porous plate. Compressed air is admitted to chambers below the porous plates, and fine bubbles rise from the upper surfaces of the plates; currents of liquid upwards over the porous plates and downwards between the depressions are thus produced, carrying with them the suspended solids and bacteria. The sewage is admitted at the bottom of one end of the tank and flows over a wall at the other end. After aeration is completed, the liquid is allowed to stand till clear, when the clarified liquid is run off and the solids are allowed to remain; fresh sewage is then admitted at once and the aeration recommenced.—J. H. J.

*Coagulant for water purification; Process for making a*—. A. Jacobson, Council Bluffs, Iowa. U.S. Pat. 1,137,005, April 27, 1915. Date of appl., April 27, 1914.

A COAGULANT consisting of ferrous sulphate and a soluble basic aluminium sulphate,  $Al_2(OH)_2(SO_4)_3$ , is prepared by passing a solution of aluminium sulphate over metallic iron.—J. H. J.

*Disinfectants for mouth-washes; Preparation of easily soluble, stable*—. E. Langer. Ger. Pat. 281,148, Nov. 28, 1911.

A MIXTURE of sodium fluoride or silicofluoride, or both of these, with tartaric acid and excess of sodium carbonate or bicarbonate is formed into tablets. Succinic acid may be used in addition to tartaric acid, and small quantities of ethereal or other aromatic substances and of soap may be incorporated with the mixture with the aid of alcohol.—A. S.

*Formaldehyde; Production of*—*from its polymers*. G. A. Ranft. Ger. Pat. 281,553, July 4, 1911.

To generate formaldehyde vapour for disinfecting purposes, polymerised formaldehyde, e.g., trioxymethylene, is treated with water and an alkali or alkaline-earth chloride or a mixture of these, and heated in an open vessel, or hot water or steam may be passed through the mixture: 30 grms. of trioxymethylene is quantitatively depolymerised by a boiling solution of 20 grms. of calcium chloride in 100 grms. of water.—A. S.

*Disinfecting and preserving agent*. Grubenholzimpregnierung Ges. m. b. H. Ger. Pat. 281,842, March 14, 1914.

A DISINFECTING solution, suitable for wood impregnation and other purposes, is obtained by adding alkali nitrite to an aqueous solution of mercuric chloride; other soluble salts not capable of decomposing alkali nitrite may also be added.

The corrosive action of a 0.1–0.2% solution of mercuric chloride on metals is inhibited by addition of 2% of alkali nitrite.—A. S.

*Water; Apparatus for purifying*—*by means of ozonised air*. G. Erlwein, Berlin, and C. Knips, Charlottenburg, Assignors to Siemens u. Halske A.-G., Berlin. U.S. Pat. 1,138,202, May 4, 1915. Date of appl., Nov. 15, 1910.

SEE Eng. Pat. 17,787 of 1910; this J., 1911, 442.

*Water; Method of softening*—. K. Schrempp, Karlsruhe, Germany. U.S. Pat. 1,139,378, May 11, 1915. Date of appl., Feb. 7, 1914.

SEE Fr. Pat. 468,047 of 1914; this J., 1914, 981.

*Detecting, indicating, and recording the presence and proportion of [inflammable] gas in the atmosphere of mines*. Eng. Pat. 9196. See XXIII.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Glucosides hydrolysable by emulsin; Biochemical detection of*—*in some plants of the families Papilionaceae and Scrophulariaceae*. E. Bourquelot and A. Fichtenholz. J. Pharm. Chim., 1915, 11, 219–225. (See also this J., 1901, 1244; 1912, 299, 510, 1051.)

EXTRACTS of the following plants were subjected to the successive action of invertase and emulsin:—*Cytisus Laburnum*, L., *Ononis Natrix*, L., *Psoralea bituminosa*, L., *Coronilla varia*, L., *Indigofera Leptostachya* (seeds), *Scrophularia aquatica*, L., *Linaria spuria*, Mill., *L. Elatine*, Desf., *L. cymbalaria*, Mill., *L. vulgaris*, Moench., *L. purpurea*, *Euphrasia officinalis*, L., and *Melampyrum arvense*, L. The changes in the optical rotation and cupric-reducing power produced by invertase indicated the presence of sucrose in all cases, except that with *Linaria spuria* the increase in reducing power was less than half that calculated from the change in optical rotation. All the extracts except those from *Coronilla varia* and *Melampyrum arvense* contained glucosides hydrolysable by emulsin, but no attempt was made to isolate them.—J. H. L.

*Grecian essential oils*. Perf. and Ess. Oil Rec., 1915, 6, 118–119.

AN examination of some essential oils now being distilled in Greece and shipped from Patras, has yielded the following results:—*Oil of laurel leaf*. Sp. gr., 0.915; optical rotation,  $-17^\circ$ ; refractive index ( $20^\circ$  C.), 1.4670; saponification value, 32.4; cineol content, about 25%. Soluble in 3 vols. of 80% alcohol with slight opalescence. *Oil of fennel*. Sp. gr., 0.937; opt. rotation,  $+27^\circ$ ; refr. index, 1.4980. Soluble in 2 vols. of 90% alcohol. Insol. in 80% alcohol. Does not solidify at  $0^\circ$  C. Its characters agree with those of wild bitter fennel oil. *Oil of origanum*. Sp. gr., 0.904; refr. index, 1.4892; phenols, 36%. Soluble in 2 vols. of 90% alcohol, insol. in 10 vols. of 80% alcohol. Phenols do not crystallise. *Oil of sage*. Sp. gr., 0.930; opt. rotation,  $+7^\circ$ ; refr. index, 1.4615; saponif. value, 21.2. Soluble in 3 vols. of 80% alcohol with slight opalescence. *Oil of rue*. Sp. gr., 0.856; opt. rotation,  $-1^\circ$ ; refr. index, 1.4340. Soluble in 3 vols. of 80% alcohol, insoluble in 70% alcohol. Does not solidify. Corresponds with oil of *Ruta bracteosa*.

*Solidago* [Golden-rod]; *Volatile oils of the genus*—. E. R. Miller and J. M. Moseley. J. Amer. Chem. Soc., 1915, 37, 1285–1294. (See also this J., 1915, 49.)

*Solidago rugosa*.—Distillation of the fresh plants yielded about 0.4% of light yellow oil: sp. gr. at

25°/25° C., 0.8620;  $n_D^{20} = -12.8^\circ$ ;  $n_D^{25} = 1.4813$ ; saponification value, 4.22; ditto after acetylation, 10.97. These numbers correspond to 1.47% of ester calculated as bornyl acetate and 1.67% of free alcohol as borneol. The fraction representing the greater portion of the oil consisted mainly of  $\alpha$ -pinene, while other fractions appeared to contain limonene and possibly  $\beta$ -pinene. *Solidago odora* (scented Golden-rod).—Distillation of the fresh flowering plants yielded 0.64–1.53% (generally over 1.0%) of yellowish oil with an odour of anise, slightly suggestive of safrol, and a sweetish taste: sp. gr. at 25°/25° C., 0.9310;  $n_D^{20} = +13.72^\circ$ ;  $n_D^{25} = 1.5065$ ; saponification value, 7.9; acid value, 0.63;  $\text{OCH}_3$ , 15.9%, corresponding to 3.11% of ester as bornyl acetate, 2.96% of free alcohol as borneol, and 75.89% of methylchavicol. A sample of oil distilled from cultivated dried plants, with a yield of 0.65%, showed: sp. gr. at 25°/15° C., 0.9450;  $n_D^{20} = -9.33^\circ$ ;  $n_D^{25} = 1.5140$ ; saponification value, 8.9; ditto after acetylation, 19.4; soluble in 0.4 vol. of 90% alcohol. The oil of *S. odora* contained 10–15% of terpenes, possibly another alcohol besides borneol, and small amounts of at least three volatile fatty acids and of non-volatile acid. The anise odour is due to methylchavicol, anethol being absent.—J. F. B.

*Costus root oil; Constituents of*—. F. W. Semmler and J. Feldstein. Ber., 1914, 47, 2687–2694. Z. angew. Chem., 1915, 28, Ref., 84–85.

The oil contains 20% of apotaxene,  $\text{C}_{15}\text{H}_{24}$ , an aliphatic hydrocarbon with four double linkages in the molecule; 7% of costol,  $\text{C}_{15}\text{H}_{22}\text{O}$ , a primary, bicyclic sesquiterpene-alcohol; 6% of  $\alpha$ -costene,  $\text{C}_{15}\text{H}_{22}$ , a bicyclic sesquiterpene; 6% of  $\beta$ -costene, a monocyclic sesquiterpene; 0.4% of phellandrene, and 0.1% of camphene.—A. S.

*Menthol; Melting and solidification points of*—. R. Meldrum. Chem. News, 1915, 111, 229–231.

The solidifying point of menthol determined by standard methods ranged from 37.5° to 40.2° C. Even by the method of adding menthol crystals to the melted mass and stirring until thick, the results varied by 0.8° C., according to the rapidity and duration of stirring. The fact that menthol solidifies below its normal melting point is attributed to its becoming colloidal prior to crystallisation, and variations in the colloidal condition and viscosity affect the solidifying point. The melting point by the thermometer bulb method was 42° C., and by the opacity method 42.5° C. The melting point of a mixture of menthol 80, and peppermint oil 20% was 33°–34.5° C., and the solidifying point was about 10° C. lower than that of menthol.

—C. A. M.

*Uric acid in urine; Colorimetric determination of*—. S. R. Benedict and E. H. Hitchcock. J. Biol. Chem., 1915, 20, 619–627.

A modification of a method proposed by Folin and Denis (J. Biol. Chem., 1913, 14, 95) is recommended. From 2 to 4 c.c. of the urine is treated with 5 c.c. of water and about 20 drops of a solution composed of 3% silver lactate solution, 70, magnesia mixture, 30, and concentrated ammonia, 100 c.c. The mixture is centrifuged, the liquid then decanted as completely as possible, the sediment is treated with 2 drops of a 5% potassium cyanide solution, 1 c.c. of water, and 2 c.c. of uric acid reagent, 10 c.c. of a 20% sodium carbonate solution is added, and, after about 30 seconds, the mixture is diluted to 50 c.c. and the coloration obtained compared with that produced under similar conditions from 5 c.c. of standard uric acid solution. The latter solution is prepared by dissolving 9 grms. of disodium phosphate and 1 gm.

of monosodium phosphate in 300 c.c. of hot water, filtering the solution if necessary, adding 200 mgrms. of pure uric acid and 1.4 c.c. of glacial acetic acid, and diluting the whole to 1 litre; 5 c.c. of chloroform may be added to prevent the growth of bacteria and moulds. The uric acid reagent is prepared by boiling together 100 grms. of sodium tungstate, 80 c.c. of 85% phosphoric acid, and 750 c.c. of water for an hour and a half under a reflux condenser, and diluting the cooled solution to 1 litre.—W. P. S.

*Uric acid in blood; Colorimetric determination of*—. S. R. Benedict. J. Biol. Chem., 1915, 20, 629–631.

TWENTY c.c. of the blood is added to 100 c.c. of boiling N/100 acetic acid, boiled for a moment, diluted with 200 c.c. of boiling water, and the mixture filtered; the insoluble portion on the filter is washed with 50 c.c. of hot water, and the filtrate is evaporated to about 25 c.c. This solution is rinsed into a flask, 2 c.c. of colloidal ferric hydroxide solution is added, the mixture is filtered, the filtrate evaporated to 2 c.c. and treated with ammoniacal silver-magnesium solution, and the determination carried out as described in the preceding abstract.—W. P. S.

*Arsenic in organic arsenic compounds alone or mixed with organic matters; Detection and determination of*—. L. Barthe. Bull. Soc. Pharm. Bordeaux, 1914, 310. J. Pharm. Chim., 1915, 11, 245–246.

For the characterisation of arsenic in organic compounds, Bressanin's reagent (see this J., 1912, 90, 93) is as sensitive as that of Bougault (compare this J., 1903, 231), but is less suited for distinguishing between different organic arsenic compounds, as the precipitate formed has always the same colour. The author carries out Bressanin's test by adding a few drops of a 10% solution of potassium iodide to a solution of the organic compound in sulphuric acid of sp. gr. 1.53. With acid of higher concentration free iodine is liberated, but this may be eliminated by adding a few drops of sodium bisulphite solution. In toxicological analyses the organic matter should first be destroyed by heating for 2 hours with sulphuric acid (25–30 c.c. of acid to 0.5 gm. of substance) in a flask closed by a funnel. The whole of the arsenic is then precipitable by hydrogen sulphide, although the destruction of the organic matter is not complete; no loss of arsenic occurs by volatilisation. The process is applicable to cacodylic acid, atoxyl, methylarsenic acid, salvarsan, and neo-salvarsan. With more complex compounds, such as the methylarsinate or cacodylate of antipyrine, it is advisable to remove the antipyrine by addition of soda and extraction with chloroform before the digestion with sulphuric acid.—J. H. L.

*Arsenious acid or sodium arsenate; Analysis of pills containing*—. M. Francois and E. Lasausse. J. Pharm. Chim., 1915, 11, 226–237.

THE small pills of the French Pharmacopoeia, each weighing about 0.02 gm. and containing 1 mgrm. of arsenious acid or sodium arsenate, often contain besides gum arabic, lactose, and honey, insoluble matters such as wheat starch. To determine the arsenic, 50–100 of the pills containing arsenious acid, or 100–200 of those containing sodium arsenate, are heated for 3 hours on a water-bath with 20 c.c. of nitric acid of 50° B. (sp. gr. 1.53). The liquid is then diluted with 20 c.c. of water, made alkaline with ammonia, filtered if necessary, treated with ammonia and 20 c.c. of magnesia mixture, and left for 3 days. The precipitate is collected on a double filter, dried at 100° C. for 10 hours, and weighed as  $\text{NH}_4\text{MgAsO}_4 \cdot 6\text{H}_2\text{O}$ . The filtrate is acidified and treated with hydrogen

sulphide to make sure that it contains no arsenic. The weighed precipitate is tested for phosphorus. —J. H. L.

**Mercury derivatives of aromatic amines. I. Structure of primary and secondary *p*-aminophenylmercuric compounds.** W. A. Jacobs and M. Heidelberger. *J. Biol. Chem.*, 1915, 20, 513—520.

THE presence of a primary amino-group in *p*-aminophenylmercuric acetate is proved by the fact that the substance can be diazotised and combined with phenols and tertiary amines with the production of azo-dyes, hence the monomolecular constitution.  $\text{H}_2\text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{Hg} \cdot \text{O} \cdot \text{OC} \cdot \text{CH}_3$  is held to be correct. *p*-Methylaminophenylmercuric acetate must have a similar constitution as it gives the reactions of a secondary amine, forming a nitrosamine which crystallises in drab-coloured hexagonal lamellae, m. pt. 183°—184° C. The following azo-derivatives of *p*-aminophenylmercuric acetate are described: 4-*p*-dimethylaminobenzeneazophenylmercuric acetate, brick red crystals, m. pt. 215° C.; 4-*p*-hydroxybenzeneazophenylmercuric acetate, orange crystals, m. pt. 218—219° C.; 4-*o*-*p*-dihydroxybenzeneazophenylmercuric acetate, prepared from resorcinol, is a brown powder, m. pt. 190—195° C. (with decomp.); and 1-amino-2-*p*-naphthaleneazophenylmercuric acetate-5-sulphonic acid, a brownish black powder. These substances dye silk yellow or orange shades. —G. F. M.

**Hexamethylenetetramine; Quaternary salts of —.** (I.) Substituted benzyl halides and the hexamethylenetetramine salts derived therefrom. (II.) Monohalogenacetylbenzylamines and their hexamethylenetetramine salts. W. A. Jacobs and M. Heidelberger. *J. Biol. Chem.*, 1915, 20, 659—683; 685—694.

(I.) SOME 70 new compounds are described, comprising benzyl alcohols and chlorides together with the hexamethylenetetramine derivatives obtained therefrom and from already known benzyl halides. (II.) The preparation and properties of 28 chloroacetyl- and iodoacetylbenzylamines and the quaternary salts obtained by their interaction with hexamethylenetetramine are described. The general method adopted in the preparation of the salts consisted in boiling equimolecular amounts of halide and hexamethylenetetramine with dry chloroform (5 to 7 c.c. for each gm. of base), until, after solution of the components, the quantity of product which separated reached a maximum. The product was separated, washed with dry chloroform and with acetone, and dried over sulphuric acid or calcium chloride and paraffin. The substances represent a new group of bactericides. When their aqueous solutions are allowed to stand or are warmed, formaldehyde is liberated and a product, consisting principally of the methylene compound of the corresponding primary amine, separates as a colloidal suspension, an emulsion, or a crystalline precipitate. —W. P. S.

**Amino-acids; Determination of mono-substituted amino groups of — by the formaldehyde-titration method.** A. Clementi. *Rend. della R. Acad. dei Lincei*, 1915, 24, 352. *Annali Chim. Appl.*, 1915, 3, 270—271.

EXPERIMENTS with sarcosine showed that amino-acids with monosubstituted amino groups react with formaldehyde, one mol. of the aldehyde combining with 2 mols. of the acid, with the linking-up of two atoms of nitrogen by a methylene group. Monosubstituted amino-acids behave as monobasic acids in the formaldehyde titration method (see this *J.*, 1908, 135); it is necessary to continue the titration in presence of phenolphthalein until an intense red colour is produced. —A. S.

**Salicylates; Some basic [substituted] —.** O. de Coninck and Gérard. *Comptes rend.*, 1915, 160, 627—628.

LEAD bromosalicylate,  $\text{C}_6\text{H}_4\text{Br} \cdot \text{O} \cdot \text{C}_6\text{H}_4 \cdot \text{CO}_2\text{Pb} \cdot \text{H}_2\text{O}$ , is precipitated by mixing lead acetate and barium bromosalicylate in aqueous solution; lead dibromosalicylate is prepared similarly. Basic barium chlorosalicylate is precipitated when normal barium chlorosalicylate is treated with dilute ammonia; barium iodosalicylate, barium diiodosalicylate, barium nitrosalicylate, and lead aminosalicylate, are prepared similarly. Basic lead salicylsulphonate,  $\text{C}_6\text{H}_4(\text{OH}) \cdot \text{SO}_3\text{Pb} \cdot \text{CO}_2\text{Pb} \cdot \text{H}_2\text{O}$ , is prepared by digesting the free acid with freshly precipitated lead oxide and evaporating to dryness. Anhydrous copper salicylate is obtained by treating the normal salt with excess of ether and heating the resulting monohydrate to 97°—98° C. in a current of dry nitrogen. All these salts are amorphous and practically insoluble in water and neutral solvents, but soluble in mineral acids. Upon heating, the dried salts carbonise readily whilst the moist salts yield an odour of phenol. —J. R.

***α*-Glycerophosphoric acid; Synthesis of —.** O. Bailly. *Comptes rend.*, 1915, 160, 663—666.

THE calcium salt of *α*-glycerophosphoric acid results from the interaction of sodium monoallylphosphate and a cold, dilute solution of potassium permanganate, followed by treatment with calcium chloride. The *α*- and *β*-glycerophosphates of calcium, strontium, and barium were prepared in a crystalline state by the slow evaporation of their aqueous solutions. The weights of the anhydrous salts contained in 100 grms. of their solutions were: for the *α*-salts, 4.95 at 15° C., 1.79 at 17° C., and 1.83 at 16° C., respectively; and for the *β*-salts, 1.72 at 15°, 2.35 at 12°, and 57.6 at 15° C., respectively. —E. H. T.

**Aliphatic hydrocarbons; Preparation and melting points of the higher —.** P. A. Levene, C. J. West, and J. van der Scheer. *J. Biol. Chem.*, 1915, 20, 521—531.

THE hydrocarbons were prepared in some cases by the reduction of the esters of fatty acids with sodium and alcohol by the method of Bouveault and Blanc (this *J.*, 1903, 888), converting the alcohols thus obtained into iodides, and reducing the latter with zinc and hydrochloric acid. Tetracosane and the higher paraffins were prepared by the action of magnesium on an ethereal solution of the iodides containing half the number of carbon atoms in their molecule. Melting points were determined in a sulphuric acid bath the temperature of which was increasing at the rate of about 1° C. in six seconds. The following are the most important observations recorded: Hexadecane,  $\text{C}_{16}\text{H}_{34}$ , from ethyl palmitate, m. pt. 20° C.; octadecane,  $\text{C}_{18}\text{H}_{38}$ , from ethyl stearate, m. pt. 28° C.; eicosane,  $\text{C}_{20}\text{H}_{42}$ , from erucic acid through arachidic acid and eicosyl alcohol, m. pt. 38° C.; docosane,  $\text{C}_{22}\text{H}_{46}$ , prepared from erucic acid by reduction with palladium and hydrogen to behenic acid, the ester of which was reduced to docosyl alcohol, m. pt. 73°—74° C., and then converted through the iodide into docosane, m. pt. 47° C.; tetracosane,  $\text{C}_{24}\text{H}_{50}$ , m. pt. 54° C.; hexacosane,  $\text{C}_{26}\text{H}_{54}$ , prepared from tridecyl iodide, has m. pt. 59°—60° C.; octacosane,  $\text{C}_{28}\text{H}_{58}$ , from tetradecyl iodide, has m. pt. 64°—65° C.; triacontane,  $\text{C}_{30}\text{H}_{62}$ , m. pt. 69°—70° C., dotriacontane,  $\text{C}_{32}\text{H}_{66}$ , from cetyl iodide and magnesium, m. pt. 74°—75° C.; tetratriacontane,  $\text{C}_{34}\text{H}_{70}$ , m. pt. 76°—76.5° C.; and hexatriacontane,  $\text{C}_{36}\text{H}_{74}$ , m. pt. 78.5° C. Cerane,  $\text{C}_{26}\text{H}_{54}$ , was also

prepared from ceryl alcohol, and melissane  $C_{30}H_{54}$ , from melissyl alcohol. They form glistening scales of m. pt.  $61^{\circ}$  and  $73-74^{\circ}$  C. respectively.—G. F. M.

*Pharmaceutical products of which the Patents have expired.* Chem. and Drug., March 20 and 27, April 3, 10, 17, 24, May 1, 8, 15, and 22. (See also this J., 1915, 301.)

*Lanolin.* Purified wool fat (ointment basis). Patent 4992 of 1882.

*Medinal.* Sodium diethylbarbiturate (hypnotic). Pats. 19,411 of 1901 and 2566 of 1905.

*Nutrose.* Sodium caseinate (nutrient). Pat. 22,190 of 1894. An aqueous solution of casein and sodium hydroxide or bicarbonate is evaporated to dryness in a vacuum.

*Oferin.* Phenylldihydroquinazoline (used in the form of its tannate as an appetiser and to prevent sickness). Pat. 13,343 of 1889. *o*-Nitrobenzylformanilide, obtained from *o*-nitrobenzylaniline and formic acid or from *o*-nitrobenzyl chloride and formanilide, is reduced by means of tin and hydrochloric acid, etc.

*Orphol.* Bismuth  $\beta$ -Naphtholate (internal antiseptic and astringent). Pats. 13,291 of 1892 and 15,361 of 1894.  $\beta$ -Naphthol is treated with bismuth nitrate in presence of caustic soda.

*Othoforn.* Methyl ester of *m*-amino-*p*-hydroxybenzoic acid (analgesic and antiseptic). Pats. 12,479 of 1897 and 22,106 of 1908. *m*-Nitro-*p*-hydroxybenzoic acid is reduced and the product esterified.

*Opaphor.* 50% solution of oxycamphor (substitute for morphine in cardiac dyspnea and asthma). Pat. 18,035 of 1896. Camphorquinone is reduced in acid, alkaline, or neutral solution.

*Pankreon.* Tannin-pancreatin compound (digestive). Pat. 14,414 of 1900. Pancreas juice or pancreatin is treated with tannin in aqueous or alkaline solution.

*Proponal.* Dipropylbarbituric acid (hypnotic). Pat. 1664 of 1903. Dipropylmalonic alkyl ester is condensed with urea in presence of sodium and alcohol, and the sodium salt is decomposed by acid.

*Protargol.* Silver proteinate (bactericide and antiseptic). Pat. 18,478 of 1897. Obtained from protalbumose or peptone and a silver salt.

*Pyramidon.* Dimethylaminoantipyrin (antipyretic). Pat. 7709 of 1896 and 9268 of 1897. Pyramidon salicylate is the subject of Eng. Pat. 11,010 of 1900 and the camphorate of No. 22,391 of 1900.

*Satipyrin.* Antipyrin salicylate (used in rheumatism and influenza). Pat. 10,432 of 1890. Phenylhydrazine, acetoacetic ester, and methyl salicylate are heated in presence of hydriodic acid and methyl alcohol.

*Sabaphen.* Acetaninophenylsalicylate (used in choria, neuralgia, etc.). Pats. 11,853 of 1891 and 3135 of 1892. Nitrophenylsalicylate is reduced and acetylated.

*Saloguinine.* Quinine salicylate (tasteless substitute for quinine and salicylic acid). Pats. 8165, 11,783, and 16,968 of 1901. Obtained by the action of quinine on salol, salicylide, or a salicyl halogenide.

*Solonal.* Piperazine quinate (uric acid solvent). Pat. 11,429 of 1899. A solution of equivalent quantities of piperazine and quinic acid in the minimum quantity of water is evaporated.

*Somabose.* Semi-digested protein (nutrient). Pats. 1897 and 3391 of 1898. Meat fibre, freed from fat and glue, is heated with a solution of oxalic acid or other acid for 15 hours at  $70^{\circ}$  C.; the product is neutralised with lime, boiled, and filtered, and the filtrate evaporated to dryness.

*Subkamin.* Mercuric sulphate-ethylenediamine (non-irritant disinfectant). Pat. 5981 of 1900.

An aqueous solution of mercuric sulphate and ethylenediamine is precipitated by alcohol.

*Tannalbin.* Albumin tannate (intestinal disinfectant). Pats. 6140 and 13,281 of 1896. Aqueous solutions of albumin and tannin are mixed and the precipitate is washed, dried, and heated at  $110^{\circ}$  to  $150^{\circ}$  C.

*Tannigen.* Diacetyltannin (used in diarrhoea). Pat. 1228 of 1894. A mixture of tannin, glacial acetic acid, and acetic anhydride is heated at  $130-140^{\circ}$  C. for 6 or 7 hours, the product is poured into water, and the resinous mass is washed with water until it is converted into a light yellow powder.

*Tannobromin.* Compound of dibromotannin and formaldehyde (disinfectant and astringent). Pat. 14,213 of 1900.

*Tannopin or tannon.* Hexamethylenetetramine-tannin (astringent and antiseptic). Pat. 10,790 of 1897. Prepared by the interaction of the components in presence of water.

*Tannocol.* Compound of gelatin and tannin (used in diarrhoea). Pat. 11,061 of 1898. The precipitate formed by adding 5% tannin solution to 1% gelatin solution, is dried at the ordinary temperature until its m. pt. is above  $100^{\circ}$  C., and finally dried at  $100^{\circ}$  C.

*Tolypyrin.* *p*-Tolyldimethylpyrazolone (antipyretic, and in rheumatism and neuralgia). Pat. 19,329 of 1892. *p*-Tolyldiazine is condensed with acetoacetic ester and the product methylated.

*Tolysal.* *p*-Tolyldimethylpyrazolone salicylate (antipyretic and antineuralgic). Pat. 19,329 of 1892. Tolypyrin is melted with salicylic acid on the water-bath.

*Tussol.* Antipyrine mandelate (for whooping cough). Pat. 11,774 of 1894. A mixture of antipyrin and mandelic acid is melted or a solution of the substances in alcohol, benzene, etc., is evaporated.

*Urosin.* Lithium quinate (for gout). Pat. 27,333 of 1898.

*Validol.* Menthyl valerate or a solution of menthol in menthyl valerate (carminative). Pat. 3706 of 1896.

*Veronal.* Diethylbarbituric acid (hypnotic). Pats. 1661, 1945, and 2954 of 1903; 14,014 of 1904; 5353 and 11,219 of 1905.

*Xeroform.* Bismuth tribromophenolate (internal antiseptic). Pats. 13,291 of 1892 and 15,361 of 1894. The sodium salt of tribromophenol is treated with bismuth nitrate in aqueous solution.

*Yohimbine.* Alkaloid of yohimbeh bark (aphrodisiac). Pat. 11,647 of 1900. The bark is extracted with dilute acetic acid, sodium carbonate is added to the solution, and the product is crystallised from alcohol.

*Detection of small quantities of sugar by the production of formaldehyde, and the constituents of urine which give rise to formaldehyde.* Salkowski. See XVII.

#### PATENTS.

*Unsaturated hydrocarbons containing the conjugated double bond: Purification of . . .* F. E. Matthews and E. H. Strange, London. Eng. Pat. 6897, Mar. 18, 1911.

When an unsaturated hydrocarbon with two double bonds is treated with sulphur dioxide, as described in Ger. Pat. 236,386 (this J., 1911, 1688) two sulphoxides are formed. The crystalline compound breaks up easily into sulphur dioxide and the initial hydrocarbon in a state of purity, but the amorphous compound is not easily decomposed and gives a poor yield of the hydrocarbon. If certain substances such as the halogen acids, iodine, and acid chlorides be added, the bulk of the sulphoxide formed is the crystalline compound. The details of the purification of isoprene by this method are given as an example. B. V. S.

*Civet*; Preparation of a ketone with a musk-like odour from —. E. Sack. Ger. Pat. 279,313, Jan. 3, 1912.

Civet is treated with alkali and then distilled with steam, the residue is extracted with ether or other suitable solvent, and after evaporating off the solvent, the extract is treated with a small quantity of alcohol, filtered, and the ketone isolated from the filtrate in the form of its semicarbazone (m.pt. 187° C.) or by other known methods. An alternative process consists in distilling the civet with an alkali hydroxide solution of about 38° B. (sp. gr. 1.357), the alkali concentration being kept approximately constant during the distillation, and then isolating the ketone from the oil which distils over. By this method other constituents of civet, including one with an odour of jasmine, are also recovered. The ketone, zibethone, with a musk-like odour, boils at 204°–205° C. at 17 mm. pressure, solidifies at 32.5° C., and has the composition,  $C_{17}H_{30}O$ .—A. S.

*Hydrohydrastinine and its homologues*; Preparation of —. E. Merck. Ger. Pat. 280,502, Jan. 23, 1913.

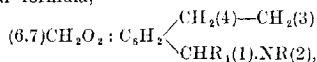
N-METHOXYMETHYLHOMOPIPERONYLAMINE or a 2- or 3-alkyl derivative of the general formula,  $(3.4)CH_2O_2 : C_6H_4 \cdot CH_2(1) \cdot CHR_1 \cdot NR_2 \cdot CH_2 \cdot O \cdot CH_3$  ( $R_1$  and  $R_2$  = hydrogen or an alkyl group) is treated with a condensing agent such as phosphorus pentoxide, whereupon methyl alcohol is eliminated and an isoquinoline ring is formed. Hydrohydrastinine is obtained from N-methoxymethylhomopiperonylamine, and the corresponding homologues from N-ethylmethoxymethyl- and N-methoxymethyl-3-methylhomopiperonylamine.—A. S.

*Tetrahydroisoquinoline derivatives* [hydrastinine derivatives]; Preparation of —. H. Decker. Ger. Pat. 281,213, Feb. 2, 1912. Addition to Ger. Pat. 270,859 (this J., 1914, 439).

N-ALKYL homologues of hydrohydrastinine or 1-alkyl or 1-aryl substitution products of hydrohydrastinine and of other N-alkyl derivatives of norhydrohydrastinine are obtained by treating 6,7-methylenedioxy-1,2,3,4-tetrahydroisoquinoline (norhydrastinine) with alkylating (with the exception of methylating) agents or by alkylating the 1-alkyl or 1-aryl substitution products of this base.—A. S.

*N-Alkyl homologues of norhydrohydrastinine and their 1-substitution derivatives*; Preparation of —. Preparation of hydrohydrastinine. H. Decker. Ger. Pats. (A) 281,546, Feb. 2, 1912, and (B) 281,547, Dec. 4, 1913. Additions to Ger. Pat. 257,138 (this J., 1913, 507).

(A) HYDROHYDRASTININE derivatives of the general formula,



in which  $R$  = alkyl,  $R_1$  = hydrogen, alkyl, or aryl, are obtained by condensing equimolecular proportions of N-monoalkyl derivatives of homopiperonylamine with aliphatic or aromatic aldehydes, and heating the products with compounds, such as hydrochloric, sulphuric, or hydrobromic acid, or phosphorus oxychloride, capable of exerting a catalytic action. Hydrohydrastinine may be obtained by heating homopiperonylamine with at least 2 mols. of formaldehyde in presence of acid, under pressure. (B) Hydrohydrastinine is prepared by heating homopiperonylamine or its N-monomethyl derivative with polymerised formaldehyde, in presence of acid, and with addition of an inert solvent or diluent, at atmospheric pressure.—A. S.

*Pyrrolidine derivatives*; Preparation of —. Chem. Fabr. auf Actien, vorm. E. Schering. Ger. Pat. 280,971, Aug. 3, 1913.

ACIDYLATED esters of pyruvic acid are condensed in non-alcoholic solution with benzaldehyde and *o*-substituted anilines, with substituted benzaldehydes and aniline or substituted anilines, with benzaldehyde and heterocyclic compounds, or with heterocyclic aldehydes and amines. The products are of value for therapeutic purposes. Examples are 1-*o*-Tolyl-2-phenyl-3-acetyl-4,5-diketopyrrolidine from *o*-toluidine, benzaldehyde in ether, and acetylpyruvic acid ester; 1,2'-methoxyphenyl-2-phenyl-3-acetyl-4,5-diketopyrrolidine from anisidine, benzaldehyde in toluene, and acetylpyruvic acid ester; 1-phenyl-2-piperonyl-3-acetyl-4,5-diketopyrrolidine from aniline, piperonal in benzene, and acetylpyruvic acid ester, and 1-*p*-tolyl-2-*p*-dimethylaminophenyl-3-acetyl-4,5-diketopyrrolidine from *p*-toluidine, *p*-dimethylaminobenzaldehyde in benzene, and acetylpyruvic acid ester.—A. S.

*Glucosides of the purine series and their derivatives*; Preparation of —. Farbenfabr. vorm. F. Bayer und Co. Ger. Pat. 281,008, Aug. 14, 1913.

METALLIC salts of purine bases or their derivatives are heated with acetylhaloglucosides or corresponding derivatives of other sugars dissolved in an inert organic solvent, and the resulting acetyl-purine-glucosides are saponified, e.g., with alcoholic ammonia. Theophylline-glucoside is obtained in this way from theophylline-silver and acetobromoglucoside. Chlorotheophylline-glucoside, theobromine-glucoside, tetra-acetyl-hydroxycaffeine-glucoside, and tetra-acetyltheophylline-galactoside have also been prepared. The new compounds exhibit diuretic properties similar to those of the purine bases from which they are derived, and possess the advantages over the latter of being more easily soluble, less poisonous, and more palatable.—A. S.

*Peroxide compound* [tetramethylenedi-urea diperoxide]; Preparation of an organic —. C. von Girsawald. Ger. Pat. 281,045, Aug. 5, 1913. Addition to Ger. Pat. 263,459.

HYDROGEN peroxide is treated, in presence of an acid, with urea and formaldehyde, instead of with hexamethylenetetramine as specified in the chief patent (this J., 1913, 992). The new compound, which is a diperoxide of tetramethylenedi-urea [ $O \cdot CH_2 \cdot NH \cdot CO \cdot NH \cdot CH_2 \cdot O$ ]<sub>2</sub>, is stable in presence of moisture and on exposure to the air. It may be used as a germicide.—A. S.

*Tetrahydropapaverine and its derivatives*; Preparation of condensation products from —. A. Pictet. Ger. Pat. 281,047, Aug. 7, 1913.

TETRAHYDROPAVERINE or one of its nuclear substitution products is condensed with aliphatic or aromatic aldehydes, preferably in the form of the corresponding acetals, in presence of a mineral acid. The divalent aldehyde radicle enters the papaverine molecule between the nitrogen atom and a carbon atom of the veratryl residue, and a second hydropyridine ring is formed. The resulting tetracyclic bases are closely allied to certain natural alkaloids, such as the corydali alkaloids, and some of them possess valuable therapeutic properties.—A. S.

*Arsenic compounds of the aromatic series*; Preparation of —. Farbenfabr. vorm. F. Bayer und Co. Ger. Pat. 281,049, July 24, 1913.

AROMATIC arsenic compounds, of value for therapeutic purposes or as intermediate products, are obtained by heating diarylamines of the benzene or naphthalene series with halogen compounds of arsenic.—A. S.

*Methylglycocyamidine (creatinine)*; Preparation of —. Farbenfabr. vorm. F. Bayer und Co. Ger. Pat. 281,051, Dec. 16, 1913.

*N-METHYLGlycocyamine* (methylguanidoacetic acid) is converted quantitatively into creatinine by heating with an organic acid.—A. S.

*2-Piperonylquinoline-4-carboxylic acid and its homologues*; Preparation of derivatives [arylates] of —. Chem. Fabr. auf Actien, vorm. E. Schering. Ger. Pat. 281,097, May 30, 1913. Addition to Ger. Pat. 252,043 (this J., 1912, 1201).

*ARYLIDES* of 2-piperonylquinoline-4-carboxylic acid and of its homologues are prepared by the known methods. Their properties are similar to those of the amides (see Ger. Pat. 277,438; this J., 1915, 249).—A. S.

*2-Piperonylquinoline-4-carboxylic acid and its derivatives*; Preparation of esters of —. Chem. Fabr. auf Actien, vorm. E. Schering. Ger. Pat. 281,136, Jan. 14, 1913.

*2-PIPERONYLQUINOLINE-4-CARBOXYLIC acid* and its derivatives are esterified with alcohols or phenols in the usual way. The esters are tasteless and exhibit therapeutic properties similar to those of phenylquinoline-4-carboxylic acid, without causing an increased separation of uric acid such as is induced by the latter.—A. S.

*2-Piperonylquinoline-4-carboxylic acid*; Preparation of homologues and substitution products of —. Chem. Fabr. auf Actien, vorm. E. Schering. Ger. Pat. 281,603, July 9, 1912.

*HOMOLOGUES* or substitution products of aniline are condensed with piperonal and pyruvic acid. The products, e.g., 6-methyl-, 6-hydroxy-, and 8-methoxy-2-piperonylquinoline-4-carboxylic acid, are tasteless.—A. S.

*Resorcinol monoacetate*; Preparation of pure, almost odourless —. Knoll und Co. Ger. Pat. 281,099, May 8, 1914.

THE product obtained by acetylating resorcinol in the usual manner is purified by treatment with slightly superheated steam *in vacuo*. Under these conditions the ester is not decomposed.—A. S.

*Catechol-o-carboxylic acid and its nuclear homologues*; Preparation of —. Farbenfabr. vorm. F. Bayer und Co. Ger. Pat. 281,214, Oct. 25, 1913.

YIELDS of up to 95% of the theoretical yields of catechol-o-carboxylic acid and its nuclear homologues are obtained by treating O-alkyl or O-alkaryl derivatives of these acids with saponifying agents, such as strong mineral acids or aluminium chloride.—A. S.

*Isolation of the selenium dyestuff corresponding to Methylene Blue*. Ger. Pat. 280,713. See IV.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Silver azide*; Photographic behaviour of —. J. Bekh. Z. wiss. Phot., 1914, 14, 105. Z. angew. Chem., 1915, 28, Ref., 93.

WHEN a film prepared with a silver azide-gelatin emulsion is exposed, a latent image is produced just as with gelatinochloride and gelatinobromide emulsions, but the azide emulsion is very much less sensitive. An unmaturred silver azide emulsion has a relatively great sensitiveness to red rays. An aqueous solution of pyrogallol is used as developer; the usual alkaline developers reduce also the unexposed azide. Silver azide is of no practical value

for the production of negatives, but printing-out papers prepared with an emulsion containing citric acid, silver nitrate, and silver azide have a sensitiveness about equal to that of celloidin paper, can be readily toned in a combined bath, and do not deteriorate on keeping.—A. S.

*Photochemical formation of electrolytes*. Boll. See VII.

PATENT.

*Colour photography*; Screens for — and for general photographic purposes. J. Rheinberg, London. Eng. Pat. 9929, April 22, 1914.

THE film which is finally to form the colour screen is covered with a resist of collodion, gelatin, albumin, casein, etc., or mixtures of these, rendered sensitive to light, before or after coating, preferably with ferric ammonium citrate or other iron salt, with which uranium salts may also be mixed. The choice of resist depends on the underlying film; both must be permeable to the dye solvent. In some cases it is preferable to interpose one or more films between the resist and the colour film. The choice of sensitiser is dependent on the dye solvent; the action of light must produce in the resist greater or less permeability to the solvent, either by altering the solubility of the sensitiser or by changing the medium itself. The composite film thus obtained is exposed behind a line or other desired screen, and then either treated with a solvent to abstract dye from unprotected parts of the underlying film, or with a dye solution to dye them. By washing and re-sensitising the resist, or by washing it off and re-coating, the process may be repeated to give any desired pattern. (See also Eng. Pat. 22,938 of 1913; this J., 1914, 1116.)—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

*Nitroglycerin-nitrocotton*; The system —. D. Chiaraviglio and O. M. Corbino. I. Condensation of nitroglycerin on nitrocotton *in vacuo* at the ordinary temperature. II. Extraction of nitroglycerin from ballistite by distillation *in vacuo* at the ordinary temperature. Rend. della R. Accad. dei Lincei, 1915, 24, 247, 361. Annali Chim. Appl., 1915, 3, 270, 271.

I. THE vapour pressure of nitroglycerin, though extremely low at the ordinary temperature, is lowered in presence of nitrocotton, and some nitroglycerin condenses on the latter, although both substances are at exactly the same temperature. It is not clear whether the phenomenon is due to imbibition or whether there is a true isothermal reaction between the two substances; if liquid nitroglycerin be dropped on to nitrocotton *in vacuo* there is a distinct development of heat.

II. If ballistite be kept, at the ordinary temperature, in a high vacuum produced by means of a Gaede pump, a considerable quantity of nitroglycerin separates from it. The separation is more rapid the greater the degree of subdivision of the ballistite, but is appreciable even when the explosive is used in its ordinary form. It is suggested that the phenomenon may prove serviceable in investigating the stability of explosives rich in nitroglycerin.—A. S.

PATENTS.

*Nitroglycerin*; Method for facilitating the separation of —. R. Weyel, Bürrigerheide, Assignor to Rheinische Dynamitfabr., Cologne, Germany. U.S. Pats. 1,138,916 and 1,138,917, May 11, 1915. Date of appl., Nov. 25, 1912.

See Eng. Pat. 14,586 of 1912; this J., 1913, 808.

*Process of forming pyroxylin bodies*. U.S. Pat. 1,135,026. See V.

## XXIII.—ANALYTICAL PROCESSES.

*Thermostat for accurate specific gravity determinations and a gas pressure regulator.* K. C. Brown and C. T. Symons. *Faraday Soc.*, May 11, 1915. [Advance proof.] 4 pages.

DETAILED particulars and a working sketch of the copper tank thermostat previously described (this J., 1914, 819) are given and also of a strong, portable gas pressure regulator capable of being rapidly set for any desired reduction of pressure. —F. SONN.

*Specific heats of liquids; Method for determining the —.* E. J. Hartung. *Faraday Soc.*, May 11, 1915. [Advance proof.] 5 pages.

A SEALED bulb of thin glass, enclosing a known weight of water and a roll of silver gauze, is brought to a steady temperature by immersion in mercury in a freezing apparatus, and is then rapidly transferred to a weighed quantity of the liquid, which is contained in a calorimeter suspended in a silvered Dewar tube. The specific heat is calculated from the fall of temperature observed, taking into account the water equivalent of the apparatus and the correction for radiation errors. The silver gauze ensures rapid melting of the ice and makes the bulb heavy enough to sink in a dense liquid. A determination may be made with about 60 c.c. of liquid. —F. SONN.

*Interferometer; Use of the — in the analysis of solutions.* L. H. Adams. *J. Amer. Chem. Soc.*, 1915, **37**, 1181—1194.

By means of the interferometer, the difference of refractive index between any two transparent liquids is measured, and if the composition of one liquid be known, that of the other can be calculated. Such comparisons and determinations can be carried out with ease, rapidly, and great precision by means of the Zeiss Water Interferometer. Light rays from a small electric lamp are converted into two interfering beams so as to form diffraction fringes observable through an eye-piece. The liquids to be compared are put into two separate compartments of a water chamber, and placed one in each path of light. The displacement of the fringes, due to the difference in refractive index, is compensated by tilting an inclined plate in the path of one beam, the tilt being measured by a micrometer screw. The instrument has been used successfully in the analysis of sea water and of colloidal solutions; it is also suitable for standardising volumetric solutions, and for analysing mixtures of sodium and potassium salts with rapidity and great accuracy. —E. H. T.

*Ammonia; Precipitant for —.* (Substitute for Nessler's reagent.) S. S. Graves. *J. Amer. Chem. Soc.*, 1915, **37**, 1171—1181.

WHEN mercuric chloride solution is made alkaline in presence of sodium chloride, a very soluble complex compound,  $\text{Na}_2\text{HgCl}_4$ , is formed, which is stable in presence of large amounts of the sodium salt. This solution precipitates quantitatively very minute amounts of ammonia, producing a bluish white cloudiness, or a white precipitate with larger quantities of ammonia. By adding lithium carbonate the tendency of the precipitated particles to coalesce is prevented, and if the reagent be employed for nephelometric measurements (in certain biochemical nitrogen determinations), a little 1% soluble starch solution is also added to keep the particles in suspension. The proportions required are: 50 c.c. cold saturated mercuric chloride solution, 15 grms. sodium chloride, 35 c.c. saturated lithium carbonate solution, and 65 c.c. water. The water used

must be free from ammonia; if a cloudiness appears, the liquid can be easily filtered after shaking with powdered talc. The reagent is more stable than that of Nessler, but fully as sensitive, precipitating ammonia from ammonium sulphate diluted to 1:1,000,000. It should be useful in water analysis, and can be applied directly to ordinary and micro-Kjeldahl determinations, rendering them less tedious and obviating errors due to distillation. —E. H. T.

*Oxalic acid; Colorimetric detection of — and of manganese.* J. F. Sacher. *Chem.-Zeit.*, 1915, **39**, 319.

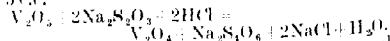
A SOLUTION of a manganese salt is treated with sodium or potassium hydroxide, and an aqueous solution of oxalic acid added, drop by drop, to the manganic hydroxide resulting from the atmospheric oxidation of the manganous hydroxide. A red coloration, which disappears on heating or on the addition of an excess of oxalic acid, is obtained, apparently through the formation of a double salt, possibly Kehrman's  $\text{K}_2\text{Mn}(\text{C}_2\text{O}_4)_3 \cdot 3\text{H}_2\text{O}$  (Ber., 1887, **20**, 1594). The test is capable of detecting 0.00025 gm. of oxalic acid in a neutral solution with a concentration of not less than 0.05%. Tannic and formic acids interfere with the coloration, and if they are present the oxalic acid should first be separated as calcium oxalate. For the detection of manganese (1:200,000), the acid solution is treated with alkali, heated, cooled, and tested with N 2 oxalic acid solution. The test is reliable even in the presence of a cobalt salt; when a large quantity of the latter is present the solution when heated yields a deposit of cobalt oxalate, while the supernatant liquid is decolorised. —C. A. M.

*Iodometric titrations [of antimony]; Secondary reactions in —.* A. Kolb. *Chem.-Zeit.*, 1915, **39**, 299—300.

ANTIMONIC acid liberates iodine from potassium iodide only in the presence of a strong acid. With acetic acid only a trace of iodine is liberated, with sulphuric acid more is set free, but concentrated hydrochloric or hydriodic acid is required to obtain the theoretical amount. The less hydrochloric acid present the more potassium iodide is required, and *vice-versa*, to obtain the same final equilibrium. The amount of iodine liberated is reduced by about 0.75% for each increase of 1°C. in the temperature. A slight excess of tartaric acid checks the separation, but ammonium, sodium, and potassium chlorides promote it. The reaction is reversible, and the iodine may be brought into combination again by antimony trioxide:  $\text{Sb}_2\text{O}_3 + 4\text{HI} \rightleftharpoons \text{Sb}_2\text{O}_3 + 2\text{H}_2\text{O} + \text{I}_2$ . To prevent secondary reactions due to atmospheric oxidation the hydrochloric acid may be saturated with carbon dioxide. —C. A. M.

*Vanadium; Determination of pentavalent — by means of sodium thiosulphate.* G. O. Oberhelman. *Amer. J. Sci.*, 1915, **39**, 530—531.

THE following procedure is recommended for the determination of vanadium:—The solution (0.1–0.2 gm.  $\text{V}_2\text{O}_5$ ) is made up to 400 c.c.; treated with 0.005 gm. of crystallised copper sulphate and 1.5 c.c. of concentrated hydrochloric acid (32% HCl), and N/10 thiosulphate run in slowly, with constant stirring, until there is an excess of 7.5 c.c.



When the colour of the solution has become sky blue after standing some time, potassium iodide (0.1 gm. per 100 c.c. of solution) is added and the excess of thiosulphate titrated with N/10 iodine in the presence of starch. When the amount of vanadate is unknown, a trial determination in

which 0.2 gm. of copper sulphate is used will give the information required for the proper adjustment of the excess of thiosulphate in an exact analysis to follow. No appreciable secondary decomposition of sodium thiosulphate takes place if the above quantities are used.—H. R. D.

*Niobium in presence of tantalum; Determination of — and some reactions of tantalum compounds.* A. G. Levy. *Analyst*, 1915, **40**, 201–217.

A METHOD is described which is based on the fluoride method of Osborne (*Amer. J. Sci.*, 1885, **30**, 328). The mixed oxides, together with 1 gm. of potassium fluoride, are dissolved in 7 c.c. of hydrofluoric acid, the solution is evaporated almost to dryness, and the residue then heated until a clear fused mass is obtained. This is transferred to a flask with the aid of 20 c.c. of water and 40 c.c. of concentrated hydrochloric acid, the niobium reduced by the addition of 10 grms. of zinc, the reduced solution diluted with a mixture of 270 c.c. of water, 10 c.c. of concentrated sulphuric acid, and 20 c.c. of a cold saturated solution of sodium phosphate, and then titrated with  $N/10$  permanganate solution (1 c.c. = 0.00834 gm.  $Nb_2O_5$ ). It is shown that large losses of tantalum are caused by dissolving tantalum oxide in hydrofluoric acid and igniting the residue obtained on evaporation to dryness, by igniting potassium tantalum fluoride with excess of potassium fluoride at a red heat, and by igniting mixtures of tantalum oxide and ammonium fluoride.—W. P. S.

*Recent progress in pyrometry.* Darling. See I.

*Fractional distillation with regulated stillheads.* Rosanoff and others. See I.

*Effect of different methods of grinding on the [determination of the] ash of coke.* Eastaugh. See IIA.

*See method for the determination of sodium and potassium in a mixture of their salts.* Okada. See VII.

*Titration of small amounts of halides.* McLean and Van Slyke. See VII.

*Oxidation of sulphides with potassium iodate.* Dean. See VII.

*Behaviour of certain hydrazine salts on decomposition by heat. [Detection of hydronitric acid (hydrazoic) acid in presence of sulphur dioxide.]* Turrentine. See VII.

*Practical methods for the determination of radium. I. Interchangeable electroscope and its use.* Lind. See VII.

*Value of the high-pressure steam test of Portland cement.* Wig and Davis. See IX.

*Determination of boron in iron.* Lindgren. See X.

*Simplified ferrous sulphate method for the determination of vanadium in steel.* Dougherty. See X.

*Technical analysis of ferrovanadium.* Heinzelmann. See X.

*Morro Velho method of assay of gold-bearing cyanide solutions.* Levy and Jones. See X.

*Rapid test for fineness of paint pigments.* Holley and Brier. See XIII.

*Use of types of tinted glass for grading rosin.* See XIII.

*Determination of sugar in bagasse.* Deerr. See XVII.

*Detection of small quantities of sugar by the production of formaldehyde.* Salkowski. See XVII.

*Determination of methyl alcohol in presence of ethyl alcohol.* Jones. See XVIII.

*Determination of fat [in animal substances, etc.].* Rosenthal and Trowbridge. See XIXA.

*Determination of chlorides in cheese.* Cornish and Golding. See XIXA.

*Determination of carbon dioxide in baking powder and carbonates.* Brubaker. See XIXA.

*Colorimetric determination of nitrous acid [in water].* Romijn. See XIXB.

*Detection and determination of arsenic in organic arsenic compounds alone or mixed with organic matters.* Barthe. See XX.

*Analysis of pills containing arsenious acid or sodium arsenate.* Francois and Lasausse. See XX.

*Colorimetric determination of uric acid in urine.* Benedict and Hitchcock. See XX.

*Colorimetric determination of uric acid in blood.* Benedict. See XX.

*Determination of mono-substituted amino groups of amino-acids by the formaldehyde titration method.* Clementi. See XX.

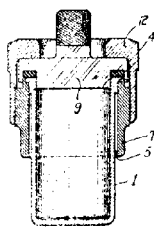
#### PATENTS.

*Gas in the atmosphere of mines; Detecting, indicating, and recording the presence and proportion of [inflammable] —.* H. R. Webster, Horsforth, Yorks. Eng. Pat. 9196, April 14, 1914.

THE porous vessel in the apparatus described in Eng. Pat. 29,094 of 1913 (this J., 1915, 131), is surrounded by a layer of cotton or glass wool, asbestos fibre, or the like, and an outer layer of caustic potash with or without quicklime, soda-lime, or calcium chloride and sodium carbonate, in order to neutralise the action of air currents and gases other than firedamp. Communication between the porous vessel and the atmosphere is afforded through a relief valve which is opened by screwing down an air-tight metal cap enclosing the porous vessel. A pressure-equalising passage leads from the underside of the diaphragm to the annular space between the cap and the porous vessel. W. F. F.

*Calorimetric apparatus. Calorimeter. Combustion device.* S. W. Parr, Urbana, Ill. U.S. Pats. (A) 1,136,359, (B) 1,136,360, and (C) 1,136,361, April 20, 1915. Dates of appl. Feb. 27, May 6, and May 6, 1914.

(A) A COMBUSTION chamber, wider at the top than at the bottom, is immersed in a water container of oval cross-section, having a rotary stirrer at one end, and a thermometer at the other end of the oval. (B) A closed combustion chamber, in which the charge is ignited, is surrounded by a perforated casing immersed in the cooling liquid and forming an air jacket. The hot air escapes through the perforations when the temperature rises, and liquid is drawn in to cool the combustion chamber rapidly when the temperature falls. (C) The chamber, 1, containing the combustible mixture, is provided with a cover, 9, and a rubber gasket, 4, which is out of contact





with the hot combustion products. The collar, 12, is screwed to the bell, 7, which partly covers the chamber, 1, leaving a narrow annular space, 5. The charge is ignited by applying a flame to the lower part of the chamber, 1, which is then immersed in the cooling liquid.—W. F. F.

[Gas] calorimeter. C. E. Lucke, New York, U.S. Pat. 1,136,884, April 20, 1915. Date of appl., June 27, 1906. Renewed April 19, 1913.

MEASURED quantities of cooling water and of gas are passed continuously, at pressures having a constant ratio, to the calorimeter, where the gas is burnt. The rise in the temperature of the water actuates an indicator.—W. F. F.

Pyrometer. A. Onwood, London. Eng. Pat. 7597, March 25, 1914.

To compensate the error in a thermocouple pyrometer due to the varying temperature of the cold junction, an auxiliary couple is placed adjacent to the cold junction of the main couple, and is connected to it, and through a resistance to the galvanometer, so that its current, when added to the current from the main couple, produces the required correction.—W. F. F.

#### XXIV.—MISCELLANEOUS ABSTRACTS.

Reaction velocity in a viscous (heterogeneous) medium. R. H. Callow, Faraday Soc., May 11, 1915. [Advance proof.] 5 pages.

EXPERIMENTS on the hydrolysis of methyl acetate in gelatin-water mixtures, with hydrochloric acid as catalyst, yielded a series of values for the velocity constant which diminished as the gelatin concentration was increased; but a large change in the viscosity involved a comparatively small change in the constant, this being only about 10% less in a set jelly than when no gelatin was present. A curve showing the relation between viscosity and velocity constant is given.—F. SODN.

Alfalfa laccase. H. H. Bunzel, J. Biol. Chem., 1915, 20, 697—706.

ALFALEA, *Medicago sativa*, does not contain an oxydase capable of oxidising quinol, and the author shows that the accelerating effect of Euler and Bolin's "laccase" obtained from *Medicago sativa* (Z. physiol. Chem., 1908, 57, 80; 1909, 61, 72) on the rate of oxidation of quinol by atmospheric oxygen is due to the alkalinity of the salts contained in such preparations.—W. P. S.

#### Trade Report.

Prohibited exports. Order in Council, May 20, 1915.

(1) CRUDE coal tar is added to the list of goods the exportation of which is prohibited to all destinations.

(2) The heading "The compounds of aniline, except aniline salt," prohibited to be exported to all destinations under the heading "Coal tar products for use in dye manufacture, including aniline oil and aniline salt," by the Order of Council, March 18th, 1915, is deleted from the list of goods the exportation of which is prohibited to all destinations abroad other than British Possessions and Protectorates.

(3) The following are added to the list of goods the exportation of which is prohibited to all destinations abroad other than British Possessions and Protectorates: Chemicals, drugs, medicinal and pharmaceutical preparations: Magnesium

chloride, oxides and salts of cobalt, oxalic acid, phosphorus. Manufactures of aluminium.

(4) The heading "India-rubber, sheet, vulcanised" (prohibited to be exported to all destinations abroad other than British Possessions and Protectorates in virtue of the Order of Council of March 18th, 1915) in the list of goods the exportation of which is prohibited to all foreign ports in Europe and on the Mediterranean and Black Seas, other than those of France, Russia (except Baltic ports), Belgium, Spain, and Portugal, is deleted.

#### Competition with Germany and Austria-Hungary.

THE Board of Trade continue to receive a very large number of inquiries for the names of sellers or buyers of articles of which the sources of supply or markets have been interfered with by the war. Special arrangements have been made in the Commercial Intelligence Branch of the Board of Trade for dealing with these inquiries, and a weekly list of articles which inquirers desire to purchase, is issued and may be obtained by United Kingdom manufacturers and exporters of British goods. British firms interested, as suppliers, in any of the goods mentioned should communicate with the Director of the Commercial Intelligence Branch of the Board of Trade, 73, Basinghall Street, London, E.C.

#### \* New Books.

[The Roman numerals in thick type refer to the similar classification of abstracts under "Journal and Patent Literature" and in the "List of Patent Applications."]

**I. Buley, J.:** Steam boilers and combustion. Narrow Crown 8vo. 220 pages. With 18 diagrams. Scott, Greenwood and Son, London. 1915. Net 4s.

**IIA. Braunkohlenindustrie,** Die deut. I. Bd. 19. u. 20. Lfg. Halle, Knapp. 1915. Each Lfg. M. 2.

**Hiscox, G. D.:** Gas, gasoline and oil engines, including producer gas and its production. Revised and enlarged by V. W. Page. 21st ed. Royal 8vo. pp. 640. Constable, London. 1915. Net 10s. 6d.

**Hobbs, D.:** Examination of hydrocarbon oils and of saponifiable fats and waxes. 8vo. Chapman and Hall. 1915. Net 21s.

**Kunerth, W.:** Illuminating power of kerosenes; comparison of the illuminating power of kerosenes used in the state of Iowa, with their physical properties. Ames, Ia., State Coll. of Agric. and Mechanic Arts. 1914. 31 pp. Illus. diagrs. 8vo.

**Webber, W. H. Y.:** Gas supply in principle and practice. With numerous original illustrations and plates. Crown 8vo. Whittaker and Co. London. 1915. Net 3s. 6d.

**Williams, R. Y., and Smith, H. E.** Mine gases and safety lamps. Urbana, Ill., Univ. of Ill. 58 pp. II. tabs. diagrs. 8vo. Pap. 1915.

**II B. Gasler, L., and Dow, J. S.:** Modern illuminants and illuminating engineering. With 294 illustrations. Med. 8vo. Whittaker and Co. London. 1915. Net 12s. 6d.

**V. Bulls, W. L.:** Development and properties of raw cotton. Ill. Cr. 8vo. pp. 234. Black. London. 1915. Net 5s.

\* Compiled by H. Grevel and Co., 33, King Street, Covent Garden, London, W.C., from whom all the works in the preceding list can be obtained.

